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1991
PERFORMANCE REPORT
DRINKING WATER ORGANICS
SECTION

MAY 1992



37/03/92



#### 1991

# PERFORMANCE REPORT DRINKING WATER ORGANICS SECTION

Report Prepared By:

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Ontario Ministry of the Environment

FEBRUARY 1992



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#### INTRODUCTION

This document represents the first publication of an annual summary of the performance of routine approved analytical methods within the laboratories of Drinking Water Organics Section.

The Drinking Water Organics Section is responsible for both qualitative and quantitative analyses of drinking, surface, river and lake waters for a wide array of organic chemicals, such as chlorinated benzenes, herbicides, pesticides, polynuclear aromatic hydrocarbons, extractable organics and purgeable organics at the part-per-trillion or part-per-billion level. In addition, analyses provided by the Section include ultra-trace quantitative analyses of environmental matrices for polychlorinated dibenzodioxins and polychlorinated dibenzofurans, most notably 2,3,7,8-tetrachlorodibenzo-p-dioxin. Also, DWO Section supports various Ministry's programs by providing characterization of unknown organic contaminants by mass spectrometry.

The major objective of the DWO Section's quality assurance program is to produce data of known quality, appropriate for a particular purpose. The quality control program is designed to detect any anomalies in the quality of the analytical results and to provide the basis for an immediate corrective action.

Within the DWO Section, the most common quality control tasks include the analyses of quality control samples, such as method blanks, fortified method blanks, samples fortified with surrogates, check calibration solutions and reference materials, and the interpretation of the resulting data. For each analytical method, the actual quality control procedures are described in detail in section 6.2 of the corresponding official method text.

This Performance Summary Report is based on the results of selected quality control samples acquired between January and December, 1991. In this report, each abstract of the analytical method is accompanied by corresponding performance charts and summary tables. Performance charts contained indicate the mean and the 99%-confidence limits for the variable presented.



METHOD CODE: OPTM-E3237A.1

METHOD TITLE: The Determination of Trihalomethanes in Water by Purge-and-Trap/Gas

Chromatography

LABORATORY: Priority Pollutants Unit

SUPERVISOR: O.W. Berg

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

Trihalomethanes are purged from an aqueous sample onto an adsorbtion trap, and subsequently, thermally desorbed onto a gas chromatographic capillary column. After separation, the organics are identified and quantified by Hall electrolytic conductivity detector.

PARAMETERS MEASURED:	LIS TEST CODE :	$\mathbf{W}$ ( $\mu$ g/L )	<b>T</b> ( μg/L )	
Chloroform	X1005J	0.5	5.0	
Bromodichloromethane	X1010J	0.2	2.0	
Dibromochloromethane	X1011J	0.2	2.0	
Bromoform	X1015J	0.2	2.0	
Total THM's	X2TTHM	0.5	5.0	

#### REPORTING FORMAT:

Results are reported in parts per billion (µg/L) rounded off to the closest increment of W and up to maximum of three significant figures.

#### QUALITY CONTROL:

The routine quality control samples are designed to verify absence of potential contamination (method blanks) and to monitor validity of calibration (calibration solutions) and the agreement with the established method precision and accuracy (laboratory replicate samples, reference material).

The results for the analysis of calibration solution and the analysis of reference material have their control limits statistically derived.

**REMARKS:** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts: Chloroform (recovery from fortified blank)

Bromodichloromethane (recovery from fortified blank) Dibromochloromethane (recovery from fortified blank)

Bromoform (recovery from fortified blank)

List of Performance Tables: Method Blanks Summary

Chloroform

Bromodichloromethane Dibromochloromethane

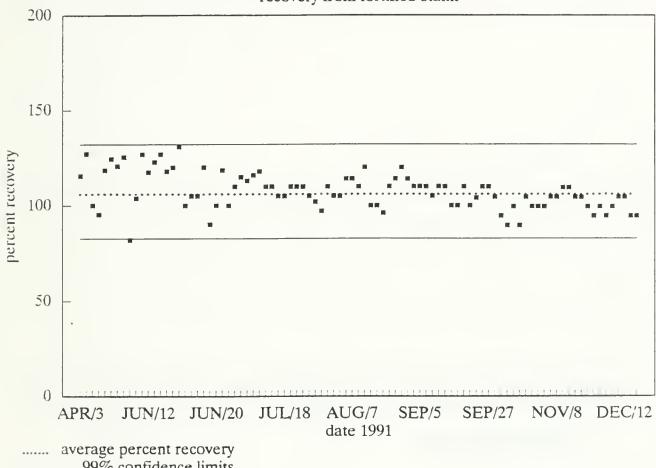
Bromoform

#### Method Blanks Summary

January 1991 - December 1991

Analyte	Number of Observations	Average Concentration ( μg/L )	Standard Deviation ( µg/L )
chloroform	106	0.05	0.23
bromodichloromethane	106	0	0
dibromochloromethane	106	0	0
bromoform	106	0	0
THM's - total	106	0.05	0.23



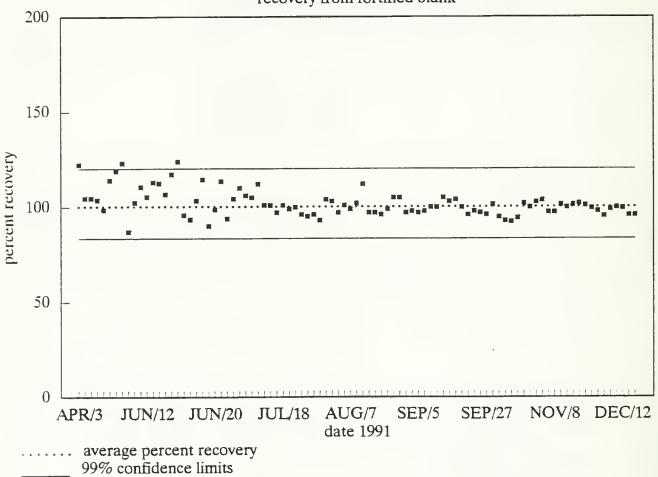


99% confidence limits

January - December 1991

Analyte	chloroform	
True Concentration	20 μg/L	
Number of Observations	91	
Within-run Rel. Standard Deviation	4% ( n=13 )	
Between-run Standard Deviation	10%	
Accuracy (% of expected)	107%	

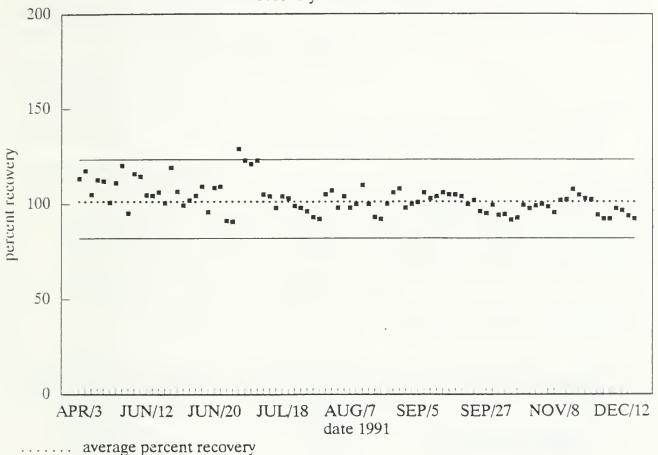
## BROMODICHLOROMETHANE recovery from fortified blank



January - December 1991

Analyte	bromodichloromethane	
True Concentration	20 μg/L	
Number of Observations	91	
Within-run Rel. Standard Deviation	5% ( n=13 )	
Between-run Standard Deviation	7%	
Accuracy (% of expected)	102%	

## DIBROMOCHLOROMETHANE recovery from fortified blank



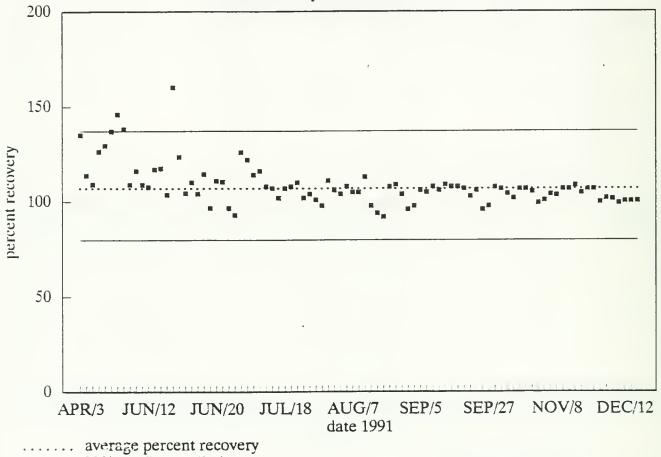
average percent recovery 99% confidence limits

Performance Summary Table

January - December 1991

Analyte	dibromochloromethane
True Concentration	20 μg/L
Number of Observations	91
Within-run Rel. Standard Deviation	5% ( n=13 )
Between-run Standard Deviation	8%
Accuracy (% of expected)	103%





average percent recovery 99% confidence limits

January - December 1991

Analyte	bromoform
True Concentration	10 µg/L
Number of Observations	91
Within-run Rel. Standard Deviation	6% ( n=13 )
Between-run Standard Deviation	11%
Accuracy (% of expected)	108%

METHOD CODE: OPOV-E3144A.1

METHOD TITLE: The Determination of Purgeable Organic Compounds in Potable and Surface

Waters by GC-FID and ECD

**LABORATORY:** Priority Pollutants Unit

SUPERVISOR: O.W. Berg

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

This method involves purge-and-trap gas chromatographic analysis with simultaneous flame ionization and electron capture detection. The volatile organics compounds are purged from aqueous phase onto a Tenax trap by a stream of helium gas. The compounds are then thermally desorbed and cryofocussed onto one-metre piece of dectivated fused silica transfer line using liquid nitrogen as the coolant. The cold trap is then rapidly heated by an electric current and the compounds are swept into the chromatographic column.

The chromatogram resulting from the FID is used for quantification, while the ECD chromatogram is used mainly for confirmation.

PARAMETERS MEASURED :	LIS TEST CODE :	<b>W</b> ( μg/L )	<b>T</b> ( μg/L )
1,1-dichloroethene	X1001P	0.1	1.0
dichloromethane	X1002P	0.5	5.0
t-1,2-dichloroethene	X1003P	0.1	1.0
1,1-dichloroethane	X1004P	0.1	1.0
chloroform	X1005P	0.1	1.0
1,1,1-trichloroethane	X1006P	0.02	0.2
1,2-dichloroethane	X1007P	0.1	1.0
carbon tetrachloride	X1008P	0.2	2.0
benzene	B2001P	0.05	0.5
1,2-dichloropropane	X1009P	0.1	1.0
trichloroethylene	X1010P	0.1	1.0
bromodichloromethane	X1011P	0.1	1.0
toluene	B2002P	0.05	0.5
1,2-dibromoethane	X2EDB	0.1	1.0
1,1,2-trichloroethane	X1012P	0.1	1.0
dibromochloromethane	X1013P	0.1	1.0
tetrachloroethene	X1014P	0.05	0.5
chlorobenzene	X2001P	0.05	0.5
ethylbenzene	B2003P	0.05	0.5
m-xylenc	B2005P	0.1	1.0
p-xylene	B2004P	0.1	1.0

### (parameters measured continued)

bromoform	X1015P	0.2	2.0
styrene	B2008P	0.05	0.5
o-xylene	B2006P	0.05	0.5
1,1,2,2-tetrachloroethane	X1016	0.2	2.0
1,4-dichlorobenzene	X2002P	0.1	1.0
1,3-dichlorobenzene	X2003P	0.1	1.0
1,2-dichlorobenzene	X2004P	0.1	1.0
total trihalomethanes	X2TTHM	0.5	5.0

#### **REPORTING FORMAT:**

Results are reported in parts per billion (µg/L) rounded off to the closest increment of W and up to maximum of three significant figures.

#### QUALITY CONTROL:

The routine quality control operations monitor absence of potential interferences (method blanks) and consistency with the predetermined method performance (fortified method blanks).

**REMARKS:** This analytical method was modified in September 1991. The detection system was replaced with dual capillary gas chromatography with dual flame ionization detectors.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB QM Office.

List of Performance Charts: all analytes described as parameters measured (recoveries from fortified

blanks)

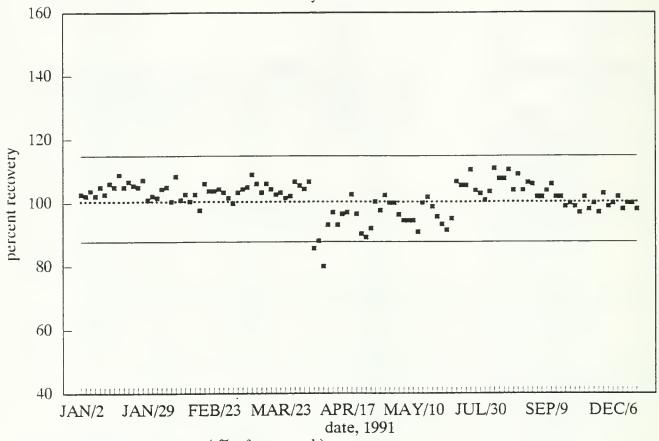
List of Performance Tables: Method Blanks Summary

all analytes described as parameters measured

Method Blanks Summary January 1991 - December 1991

	· · · · · · · · · · · · · · · · · · ·		
Analyte	Number of Observations	Average Concentration ( μg/L )	Standard Deviation ( µg/L )
1,1-dichloroethene	205	0	0
dichloromethane	205	0.40	0.93
t-1,2-dichloroethene	205	0	0
1,1-dichloroethane	205	0	0
chloroform	205	0.0007	0.0074
1,1,1-trichloroethane	205	0	0
1,2-dichloroethane	205	0	0
carbon tetrachloride	205	0	0
benzene	205	0.0038	0.0053
1,2-dichloropropane	205	0	0
trichloroethene	205	0.0004	0.0063
bromodichloromethane	205	0.0001	0.0010
toluene	205	0.0078	0.0075
1,2-dibromoethane	205	0	0
1,1,2-trichloroethane	205	0	0
dibromochloromethane	205	0	0
tetrachloroethene	205	0	0
chlorobenzene	205	0	0
ethylbenzene	205	0.0004	0.0025
m-xylene / p-xylene	205	0.0027	0.0050
bromoform	205	0	0
styrene	205	0.0027	0.0066
o-xylene	205	0.0008	0.0027
1,1,2,2-tetrachloroethane	205	0.001	0.018
1,4-dichlorobenzene	205	0.006	0.012
1,3-dichlorobenzene	205	0.004	0.011
1,2-dichlorobenzene	205	0.005	0.013
total trihalomethanes	205	0.0009	0.0074

## 1,1-DICHLOROETHENE recovery from fortified blank

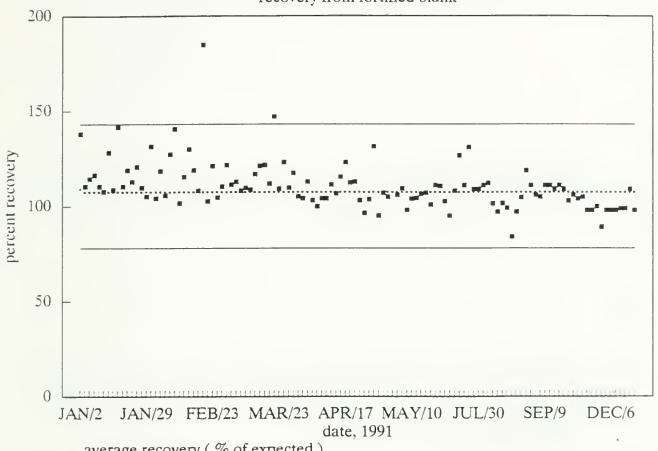


average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	1,1-dichloroethene
True Concentration	1.77 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	2.2% ( n=4 )
Between-run Standard Deviation	5.2%
Accuracy (% of expected)	101%

### **DICHLOROMETHANE** recovery from fortified blank

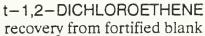


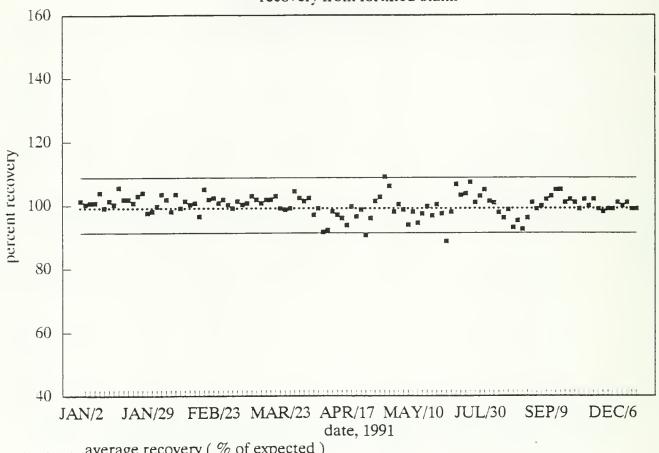
average recovery (% of expected)

99% confidence limits

January - December 1991

Analyte	dichloromethane
True Concentration	1.95 μg/L, 3.68 μg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1% ( n=4)
Between-run Standard Deviation	12%
Accuracy (% of expected)	111%



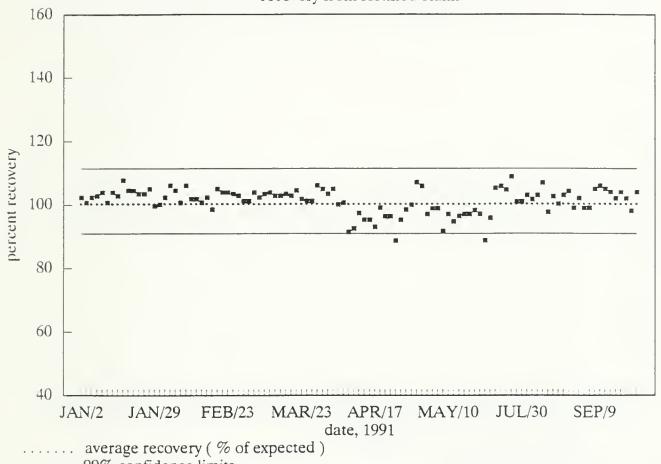


average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	t-1,2-dichloroethene
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.3% ( n=4 )
Between-run Standard Deviation	3.4%
Accuracy (% of expected)	100%

### 1,1-DICHLOROETHANE recovery from fortified blank

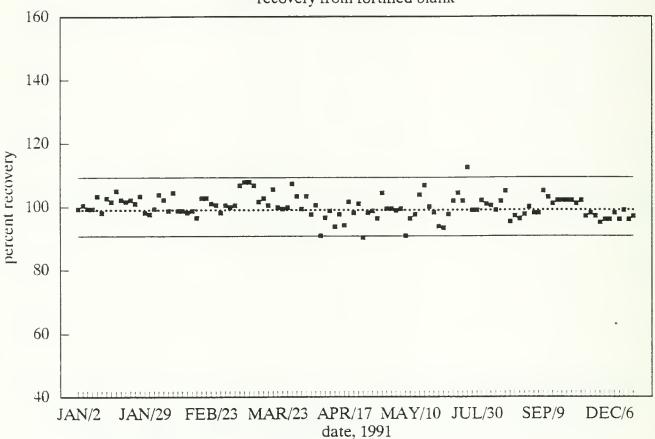


99% confidence limits

January - December 1991

Analyte	1,1-dichloroethane
True Concentration	1.84 µg/L, 3.68 µg/L
Number of Observations	107
Within-run Rel. Standard Deviation	1.1% ( n=4 )
Between-run Standard Deviation	4.0%
Accuracy (% of expected)	101%



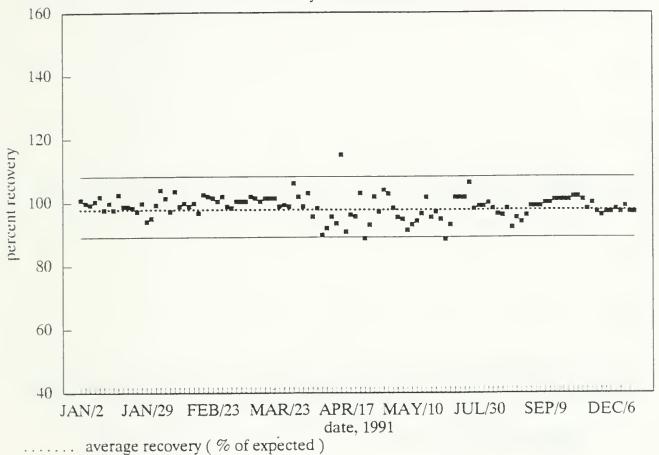


..... average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	chloroform
True Concentration	1.77 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% ( n=4 )
Between-run Standard Deviation	3.6%
Accuracy (% of expected)	100%

## 1,1,1-TRICHLOROETHANE recovery from fortified blank

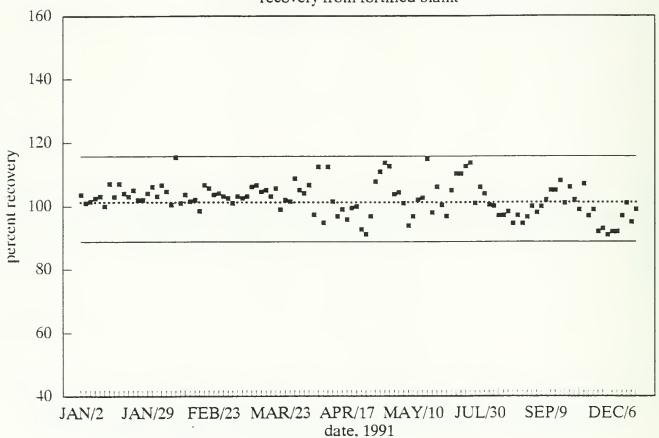


99% confidence limits

January - December 1991

Analyte	1,1,1-trichloroethane
True Concentration	1.89 μg/L, 3.68 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% ( n=4 )
Between-run Standard Deviation	3.7%
Accuracy (% of expected)	98.6%

## TETRACHLOROMETHANE recovery from fortified blank

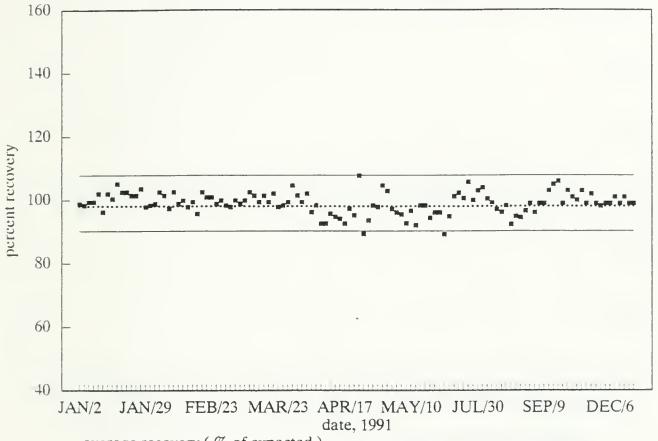


...... average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	tetrachloromethane
True Concentration	1.94 μg/L, 3.68 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.7% ( n=4 )
Between-run Standard Deviation	5.2%
Accuracy (% of expected)	102.2%

BENZENE recovery from fortified blank

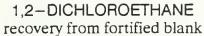


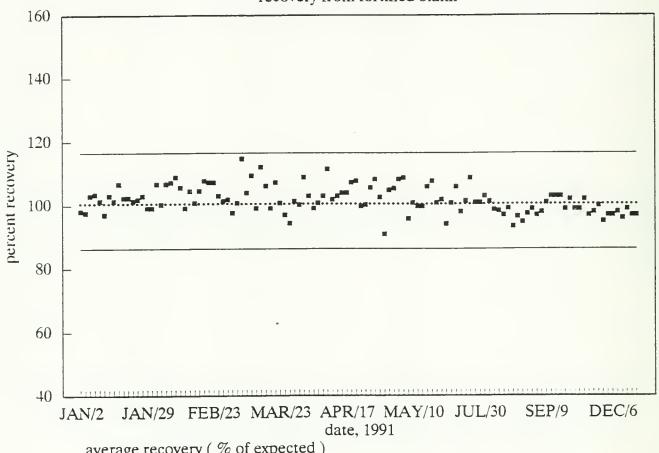
..... average recovery (% of expected) 99% confidence limits

Performance Summary Table

January - December 1991

Analyte	benzene
True Concentration	1.90 μg/L, 3.68 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.3% ( n=4 )
Between-run Standard Deviation	3.4%
Accuracy (% of expected)	99%



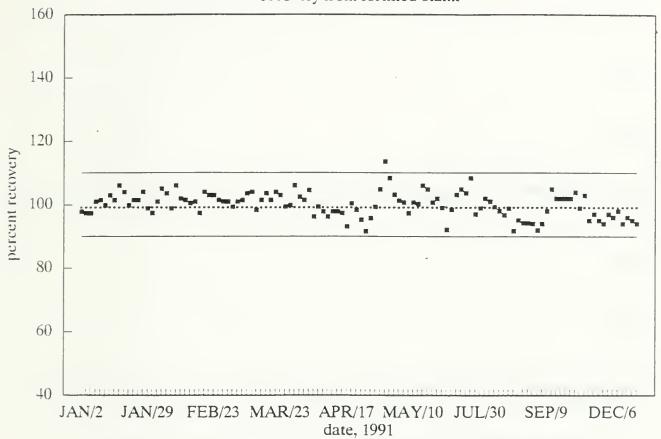


...... average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	1,2-dichloroethane
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.0% ( n=4 )
Between-run Standard Deviation	5.8%
Accuracy (% of expected)	101.5%

## TRICHLOROETHENE recovery from fortified blank

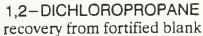


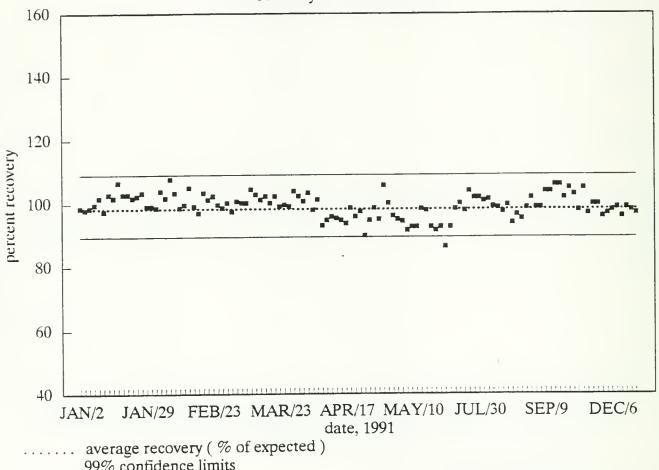
average recovery (% of expected) 99% confidence limits

Performance Summary Table

January - December 1991

Analyte	trichloroethene
True Concentration	1.92 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% ( n=4 )
Between-run Standard Deviation	3.9%
Accuracy (% of expected)	100%



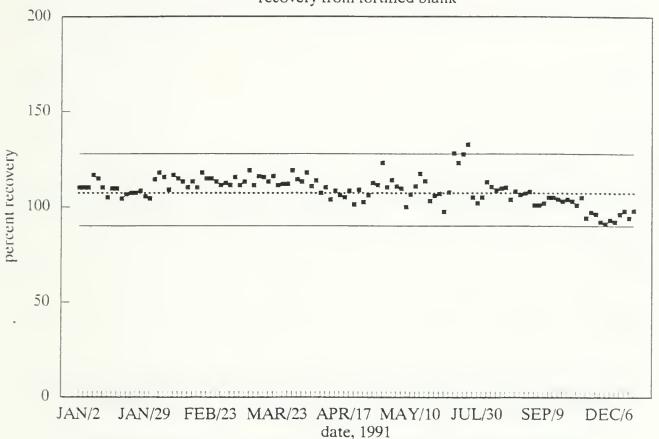


99% confidence limits

January - December 1991

Analyte	1,2-dichloropropane
True Concentration	1.87 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	0.9% ( n=4 )
Between-run Standard Deviation	3.7%
Accuracy (% of expected)	99%

## BROMODICHLOROMETHANE recovery from fortified blank

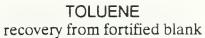


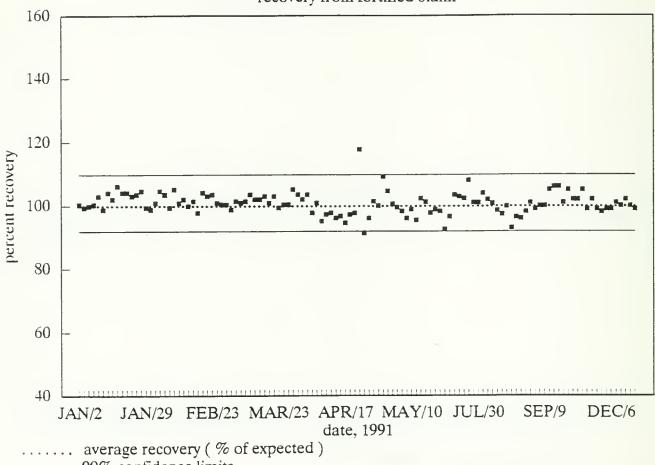
.... average recovery (% of expected)
99% confidence limits

Performance Summary Table

January - December 1991

Analyte	bromodichloromethane
True Concentration	1.70 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% ( n=4 )
Between-run Standard Deviation	7.4%
Accuracy (% of expected)	110%



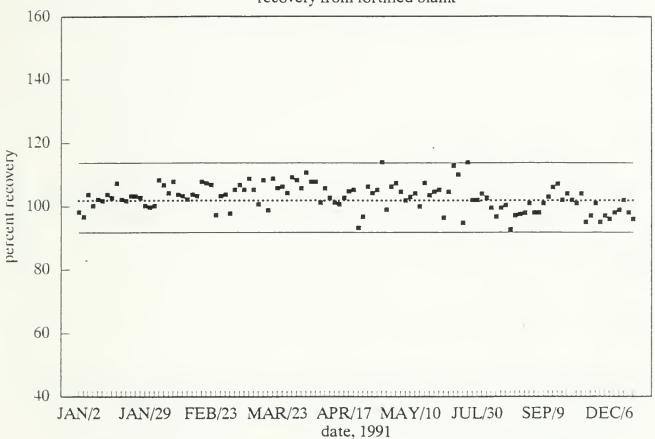


99% confidence limits

January - December 1991

Analyte	toluene
True Concentration	1.90 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.5% ( n=4 )
Between-run Standard Deviation	3.5%
Accuracy (% of expected)	101%

## 1,1,2-TRICHLOROETHANE recovery from fortified blank

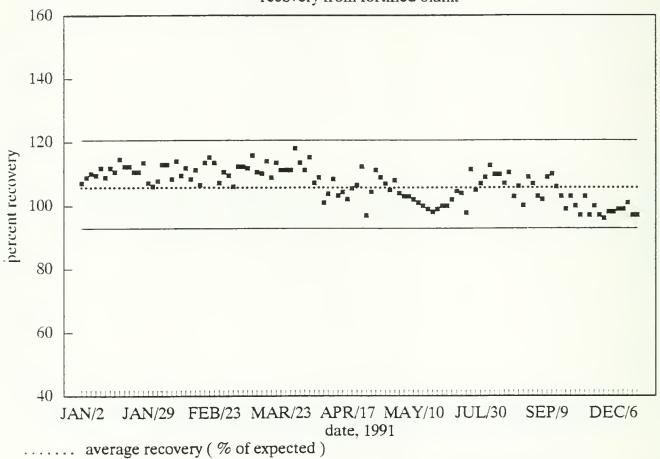


..... average recovery (% of expected) 99% confidence limits

January - December 1991

Analyte	1,1,2-trichloroethane
True Concentration	2.01 μg/L, 3.68 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% ( n=4 )
Between-run Standard Deviation	4.2%
Accuracy (% of expected)	103%

### **TETRACHLOROETHENE** recovery from fortified blank

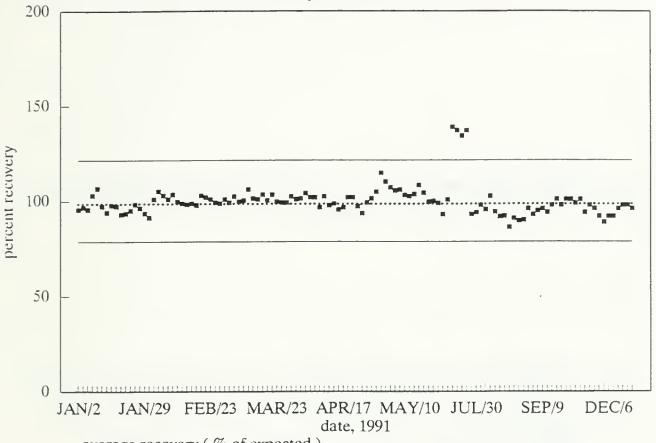


99% confidence limits

January - December 1991

Analyte	tetrachloroethene
True Concentration	1.74 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.7% ( n=4 )
Between-run Standard Deviation	5.4%
Accuracy (% of expected)	107%

## DIBROMOCHLOROMETHANE recovery from fortified blank

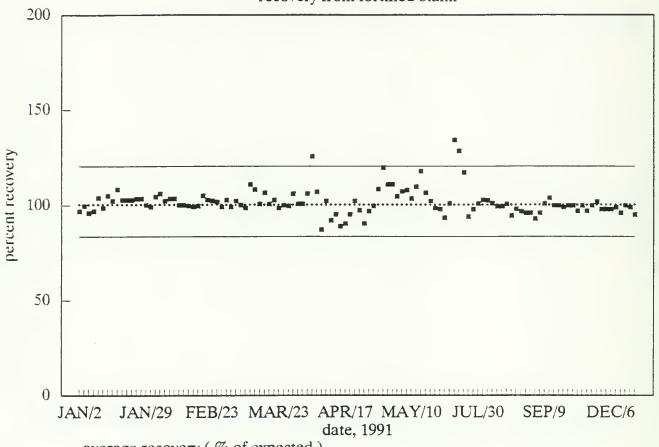


average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	dibromochloromethane
True Concentration	1.90 µg/L, 6.44 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.9% ( n=4 )
Between-run Standard Deviation	8.2%
Accuracy (% of expected)	100.1%

1,2-DIBROMOETHANE recovery from fortified blank

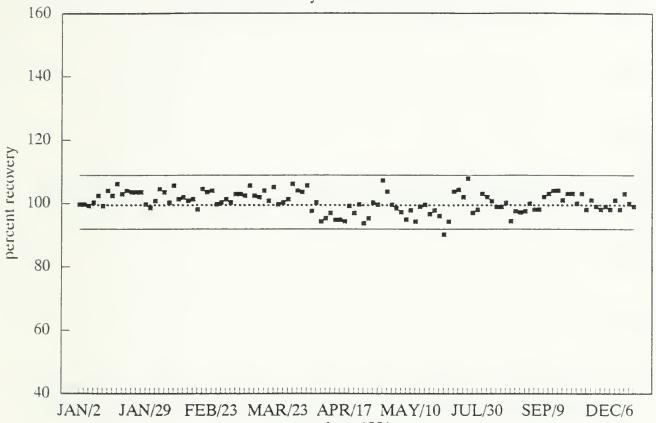


...... average recovery (% of expected) 99% confidence limits

January - December 1991

Analyte	1,2-dibromoethane
True Concentration	1.85 μg/L, 5.52 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% ( n=4 )
Between-run Standard Deviation	6.8%
Accuracy (% of expected)	102%

### **CHLOROBENZENE** recovery from fortified blank



date, 1991

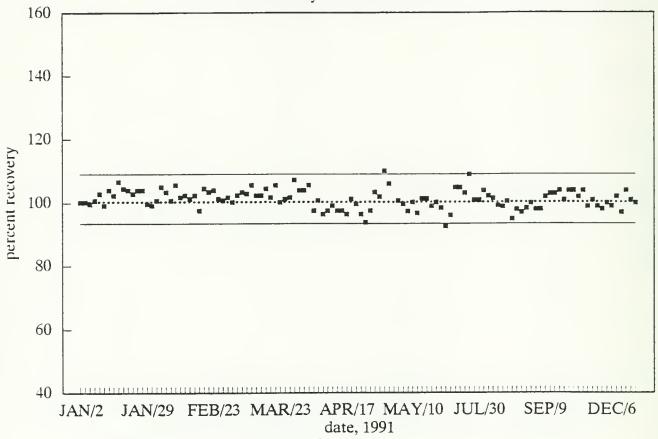
average recovery (% of expected) 99% confidence limits

Performance Summary Table

January - December 1991

Analyte	chlorobenzene
True Concentration	1.86 μg/L, 4.6 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% ( n=4 )
Between-run Standard Deviation	3.3%
Accuracy (% of expected)	100.4%

## ETHYLBENZENE recovery from fortified blank

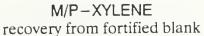


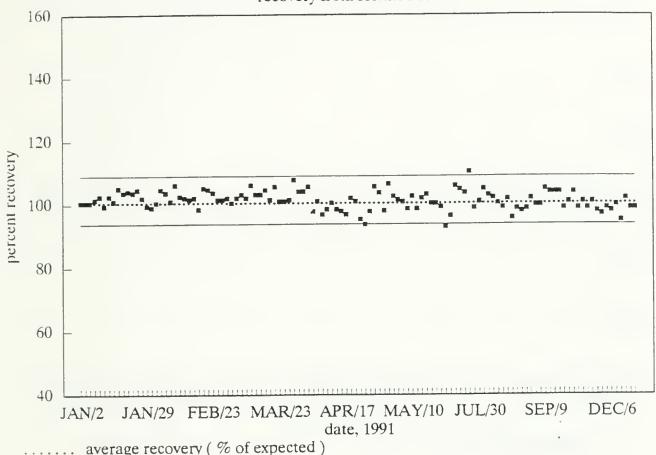
..... average recovery (% of expected)
99% confidence limits

Performance Summary Table

January - December 1991

Analyte	ethylbenzene
True Concentration	1.87 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% ( n=4 )
Between-run Standard Deviation	3.0%
Accuracy (% of expected)	101%





average recovery (% of expected)

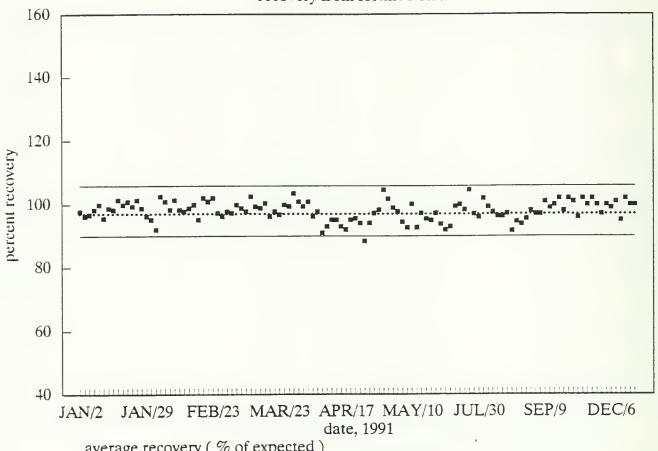
99% confidence limits

Performance Summary Table

January - December 1991

Analyte	m/p-xylene
True Concentration	1.94 μg/L, 3.68 μg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.3% ( n=4 )
Between-run Standard Deviation	2.9%
Accuracy (% of expected)	101%

O-XYLENE recovery from fortified blank

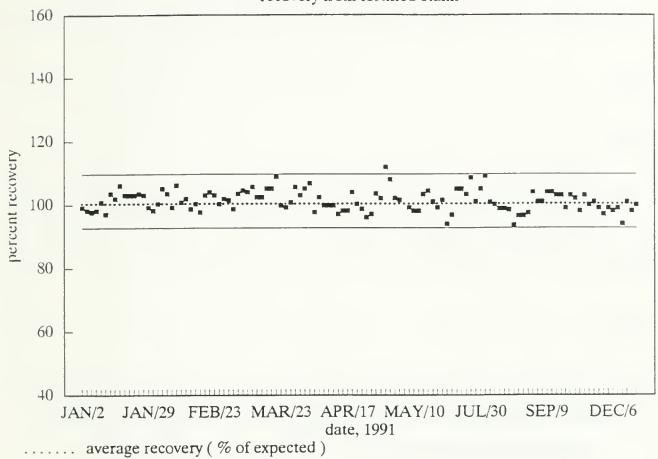


average recovery (% of expected)
99% confidence limits

January - December 1991

Analyte	o-xylene
True Concentration	1.91 μg/L, 3.68 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.5% ( n=4 )
Between-run Standard Deviation	3.1%
Accuracy (% of expected)	98%





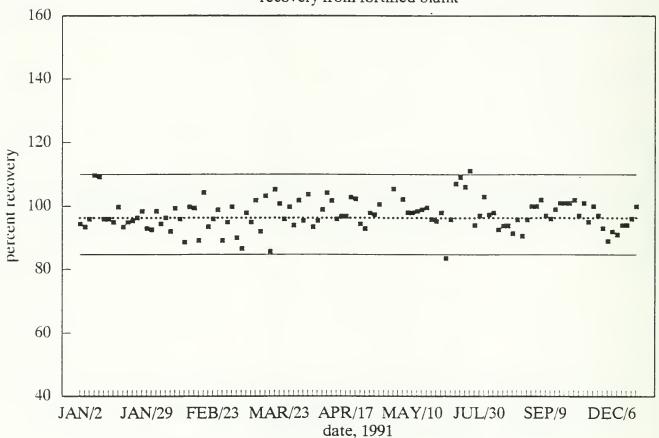
average recovery (% of expected)
99% confidence limits

Performance Summary Table

January - December 1991

Analyte	styrene
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.8% ( n=4 )
Between-run Standard Deviation	3.3%
Accuracy (% of expected)	101%

## BROMOFORM recovery from fortified blank



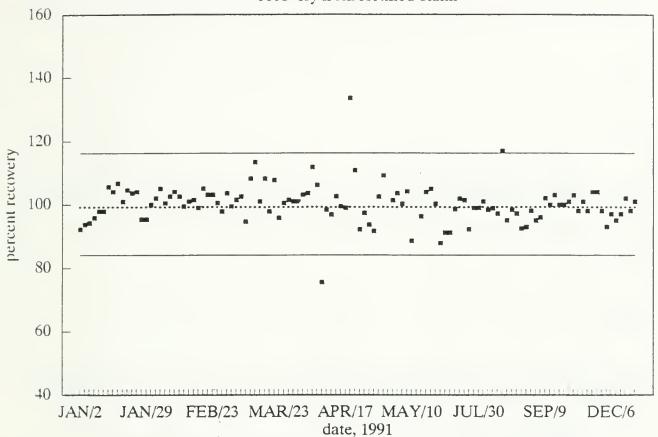
average recovery (% of expected)
99% confidence limits

Performance Summary Table

January - December 1991

Analyte	bromoform
True Concentration	2.04 μg/L, 7.36 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	4.8% ( n=4 )
Between-run Standard Deviation	4.9%
Accuracy (% of expected)	97%

## 1,1,2,2—TETRACHLOROETHANE recovery from fortified blank

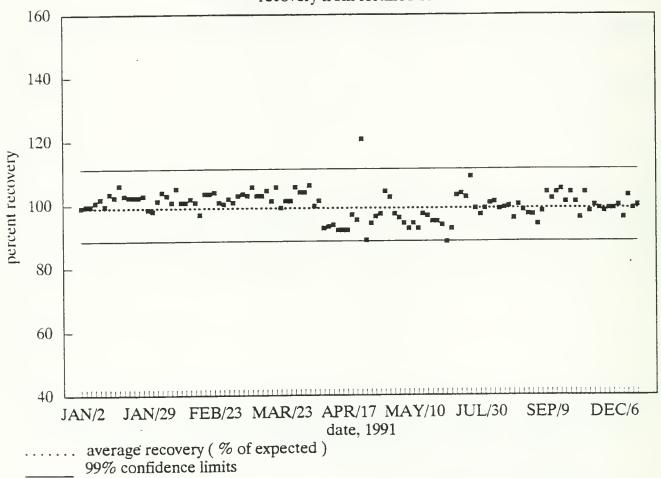


..... average recovery ( % of expected ) 99% confidence limits

January - December 1991

Analyte	1,1,2,2-tetrachloroethane
True Concentration	1.93 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	2.6% ( n=4 )
Between-run Standard Deviation	7.0%
Accuracy (% of expected)	99.5%

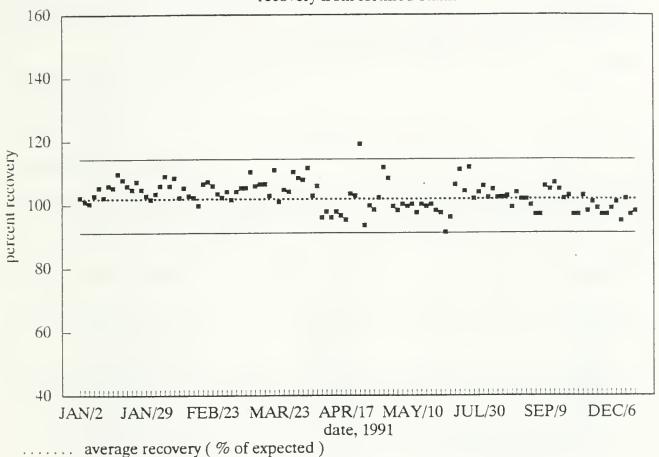
## 1,3-DICHLOROBENZENE recovery from fortified blank



January - December 1991

Analyte	1,3-dichlorobenzene
True Concentration	1.86 μg/L, 5.52 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.0% ( n=4 )
Between-run Standard Deviation	4.4%
Accuracy (% of expected)	100%



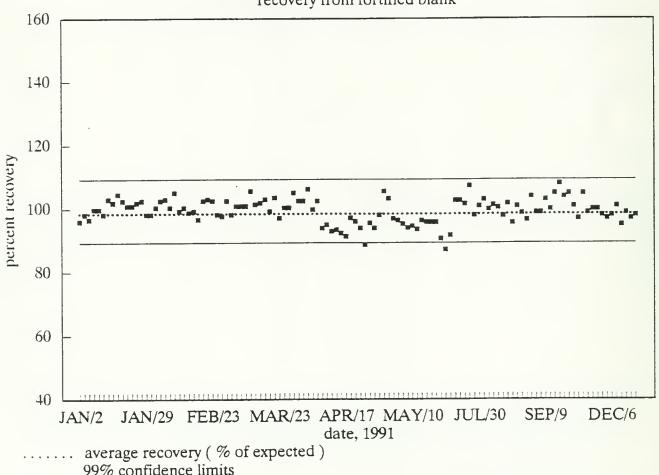


99% confidence limits

January - December 1991

Analyte	1,4-dichlorobenzene
True Concentration	1.59 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.0% ( n=4 )
Between-run Standard Deviation	4.5%
Accuracy (% of expected)	103%

1,2-DICHLOROBENZENE recovery from fortified blank



99% confidence limits

January - December 1991

Analyte	1,2-dichlorobenzene
True Concentration	1.88 μg/L, 5.52 μg/L
Number of Observations	118
Within-run Rel. Standard Deviation	0.9% ( n=4 )
Between-run Standard Deviation	3.9%
Accuracy (% of expected)	99.3%

METHOD CODE: OWOC-E3120A.1

METHOD TITLE: The Determination of Organochlorine Pesticides, Polychlorinated Biphenyls and

Other Chlorinated Organic Compounds in Water by GC-ECD

LABORATORY: Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

Samples are extracted with solvent; the extract is dried, concentrated, cleaned-up on Florisil, and reconcentrated prior to analysis by dual column capillary gas chromatography with dual electron capture detection.

PARAMETERS MEASURED :	LIS TEST CODE :	W ( ng/L )	T ( ng/L )
hexachloroethane	X2HCE	1	10
1,3,5-trichlorobenzene	X2135	5	50
1,2,4-trichlorobenzene	X2124	5	50
1,2,3-trichlorobenzene	X2123	5	50
hexachlorobutadiene	X1HCBD	1	10
2,4,5-trichlorotoluene	X2T245	5	50
2,3,6-trichlorotoluene	X2T236	5	50
1,2,3,5-tetrachlorobenzene	X21235	1	10
1,2,4,5-tetrachlorobenzene	X21245	1	10
1,2,3,4-tetrachlorobenzene	X21234	1	10
$\alpha$ ,2,6-trichlorotoluene	X2T26A	5	50
pentachlorobenzene	X2PNCB	1	10
hexachlorocyclopentadiene	X1HCCP	5	50
hexachlorobenzene	X2HCB	1	10
heptachlor	PIHEPT	1	10
aldrin	P1ALDR	1	10
p,p'-DDE	P1PPDE	1	10
α-BHC	P1BHCA	1	10
в-внс	P1BHCB	1	10
ү-ВНС	P1BHCG	1	10
α-chlordane	P1CHLA	2	20
γ-chlordane	P1CHLG	2	20
oxychlordane	P1OCHL	2	20
o,p'-DDT	PIOPDT	5	50
p.p'-DDD	P1PPDD	5	50
p,p'-DDT	PIPPDT	5	50
methoxychlor	PIDMDT	5	50

#### ( parameters measured continued )

heptachlor epoxide	P1HEPE	1	10
endosulfan I	P1END1	2	20
dieldrin	P1DIEL	2	20
endrin	PIENDR	5	50
endosulfan II	P1END2	5	50
endosulfan cyclic sulfate	PIENDS	5	50
mirex	P1MIRX	5	50
total PCB's	P1PCBT	20	200
octachlorostyrene	X2OCST	1	10
toxaphene	PITOX	500	5 000

#### **REPORTING FORMAT:**

Results are reported in parts per trillion ( ng/L ) rounded off to the closest increment of W and up to maximum of two significant figures.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS:** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality

Management Office.

List of Performance Charts: Hexachlorobenzene (recovery from fortified blank)

1,3,5-Trichlorobenzene (recovery from fortified blank)
Hexachlorobutadiene (recovery from fortified blank)

Mirex ( recovery from fortified blank )
Total PCB ( recovery from fortified blank )

List of Performance Tables: Method Blanks Summary

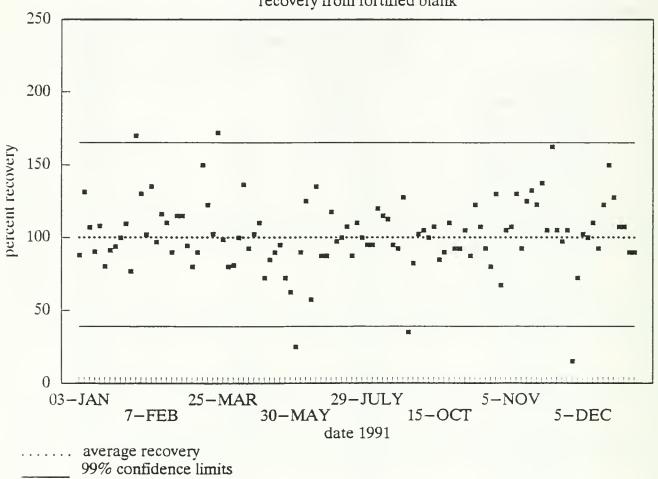
Hexachlorobenzene 1,3,5-Trichlorobenzene Hexachlorobutadiene

Mirex Total PCB Method Blanks Summary

January 1991 - December 1991

	Number of	Average Concentration	Standard Deviation
Analyte	Observations	( ng/L )	( ng/L )
hexachloroethane	136	1.5	3.6
1,3,5-trichlorobenzene	136	0	0
1,2,4-trichlorobenzene	136	0	0
1,2,3-trichlorobenzene	136	0	0
hexachlorobutadiene	136	0.04	0.25
2,4,5-trichlorotoluene	136	0.1	1.1
2,3,6-trichlorotoluene	136	0	0
1,2,3,5-tetrachlorobenzene	136	0	.0
1,2,4,5-tetrachlorobenzene	136	0.04	0.051
1,2,3,4-tetrachlorobenzene	136	0.05	0.60
α,2,6-trichlorotoluene	136	0	0
pentachlorobenzene	136	0	0
hexachlorocyclopentadiene	136	0.7	3.0
hexachlorobenzene	136	0	0
heptachlor	136	0	0
aldrin	136	0	0
p,p'-DDE	136	0.007	0.085
α-ВНС	136	0.39	0.74
в-внс	136	0.02	0.26
у-ВНС	136	0.007	0.085
α-chlordane	136	0	0
γ-chlordane	136	0	0
oxychlordane	136	0	0
o,p'-DDT	136	0	0
p,p'-DDD	136	0	0
p,p'-DDT	136	0	0
methoxychlor	136	0	0
heptachlor epoxide	136	0	0
endosulfan I	136	0	0
dieldrin	136	0	0
endosulfan II	136	0	0
endosulfan cyclic sulfate	136	0.03	0.34
mirex	136	0	0
total PCB's	136	1	13
octachlorostyrene	136	0	0
toxaphene	136	0	0

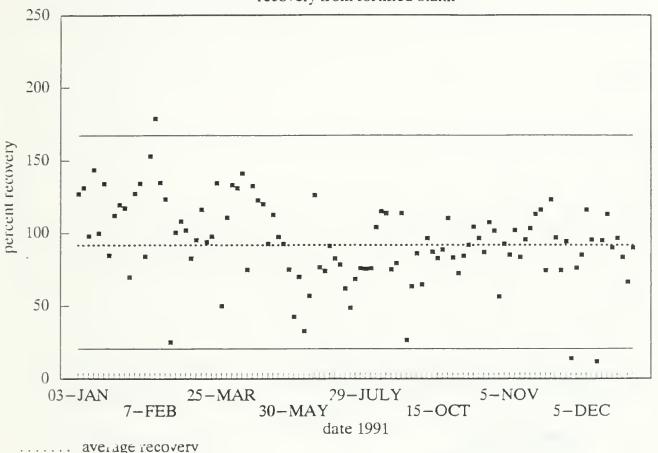




January - December 1991

Analyte	hexachlorobenzene
True Concentration	10 ng/L; 20 ng/L
Number of Observations	109
Within-run Standard Deviation	not available
Between-run Standard Deviation	24%
Accuracy (% of expected)	102%



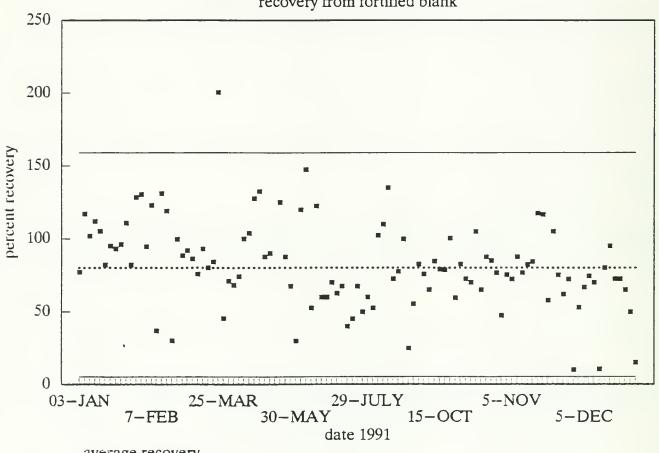


Performance Summary Table

January - December 1991

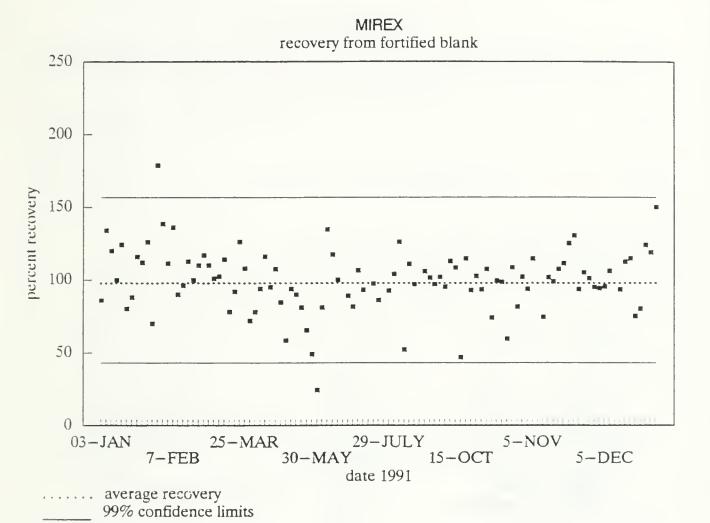
Analyte	1,3,5-trichlorobenzene	
True Concentration	10 ng/L; 20 ng/L	
Number of Observations	109	
Within-run Standard Deviation	not available	
Between-run Standard Deviation	28%	
Accuracy (% of expected)	94%	





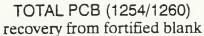
January - December 1991

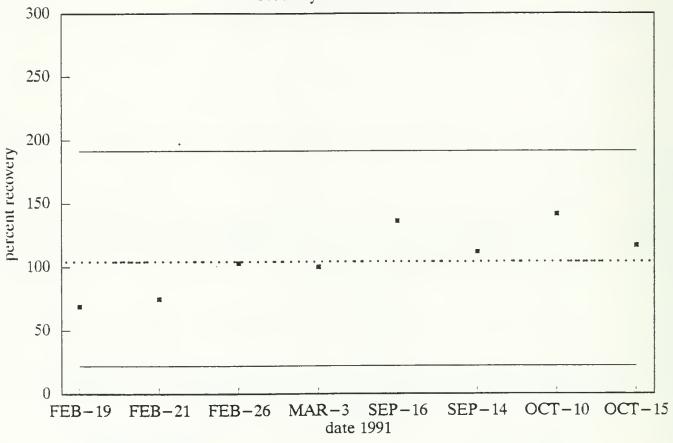
Analyte	hexachlorobutadiene
True Concentration	10 ng/L; 20 ng/L
Number of Observations	108
Within-run Standard Deviation	not available
Between-run Standard Deviation	30%
Accuracy (% of expected)	82%



January - December 1991

Analyte	mirex
True Concentration	50 ng/L; 100 ng/L
Number of Observations	105
Within-run Standard Deviation	not available
Between-run Standard Deviation	22%
Accuracy (% of expected)	98%





January - December 1991

Analyte	total PCB ( 1254/1260)	
True Concentration	200 ng/L	
Number of Observations	8	
Within-run Standard Deviation	not available	
Between-run Standard Deviation	24%	
Accuracy (% of expected)	107%	

METHOD CODE: OWCP-B-E3119A.1

**METHOD TITLE:** The Determination of Chlorophenols and Phenoxyacid Herbicides in Water by

Using Solid Phase Extraction and GC-ECD

**LABORATORY:** Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

The aqueous sample is aspirated through C-18 bonded porous silica cartridge. The cartridge is eluted with a small volume of solvent. The eluate is methylated and the 2,4-D type herbicides and chlorophenols are determined as the corresponding methyl esters and ethers by dual capillary gas chromatography with electron capture detection.

PARAMETERS MEASURED:	LIS TEST CODE :	W ( ng/L )	T ( ng/L )
2,4,6-trichlorophenol	X3246	20	200
2,4,5-trichlorophenol	X3245	100	1 000
2,3,4-trichlorophenol	X3234	100	1 000
2,3,5,6-tetrachlorophenol	X32356	10	100
2,3,4,5-tetrachlorophenol	X32345	20	200
pentachlorophenol	Х3РСРН	10	100
Dicamba	P3D1CA	50	500
2,4-dichlorophenoxypropanoic a	cid P324DP	100	1 000
2,4-dichlorophenoxyacetic acid	P324D	100	1 000
Silvex	P3SILV	20	200
2,4,5-trichlorophenoxyacetic aci	d P3245T	50	500
2,4-dichlorophenoxybutyric acid	l P324DB	200	2 000
Picloram	P3PICL	100	1 000

### **REPORTING FORMAT:**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

#### QUALITY CONTROL:

Quality control samples included in the run format are method blanks, fortified method blanks and calibration check solution.

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS**: In addition to the intra-laboratory method control, the performance of the method was

examined through performance audit samples program organized by LSB Quality

Management Office.

List of Performance Charts: 2,4,6-Trichlorophenol (recovery from fortified blank)

2,4-Dichlorophenoxyacetic Acid (recovery from fortified blank)

Silvex (recovery from fortified blank)

List of Performance Tables: Method Blanks Summary

2,4,6-Trichlorophenol

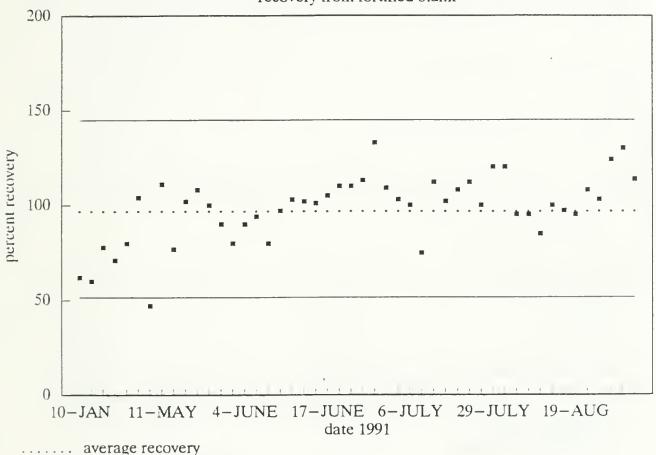
2,4-Dichlorophenoxyacetic Acid

Silvex

Method Blanks Summary January 1991 - December 1991

mediod Bianks Sammary	January 1771	Beccinoer 1991	
Analyte	Number of Observations	Average Concentration ( ng/L )	Standard Deviation ( ng/L )
2,4,6-trichlorophenol	24	0	0
2,4,5-trichlorophenol	24	0	0
2,3,4-trichlorophenol	24	0	0
2,3,5,6-tetrachlorophenol	24	0	0
2,3,4,5-tetrachlorophenol	24	0	0
pentachlorophenol	24	6	42
Dicamba	24	0	0
2,4-dichlorophenoxypropanoic acid	24	0	0
2,4-dichlorophenoxyacetic acid	24	0	0
Silvex	24	0	0
2,4,5-trichlorophenoxyacetic acid	24	0	0
2,4-dichlorophenoxybutyric acid	24	0	0
Picloram	24	0	0
1		1	

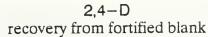


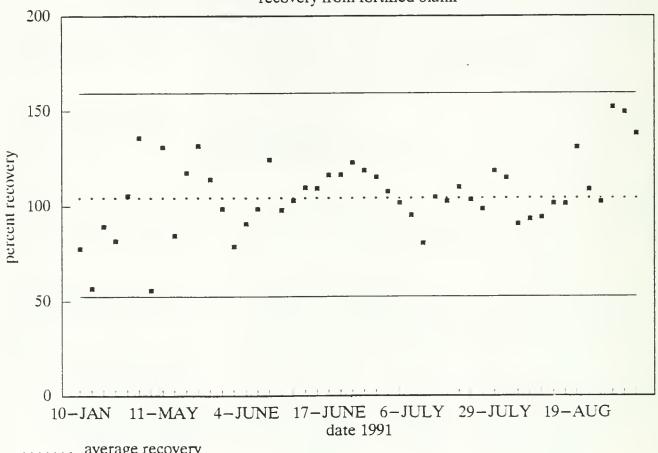


Performance Summary Table

January - December 1991

Analyte	2,4,6-trichlorophenol
True Concentration	100 ng/L
Number of Observations	48
Within-run Standard Deviation	not available
Between-run Standard Deviation	17%
Accuracy (% of expected)	98%

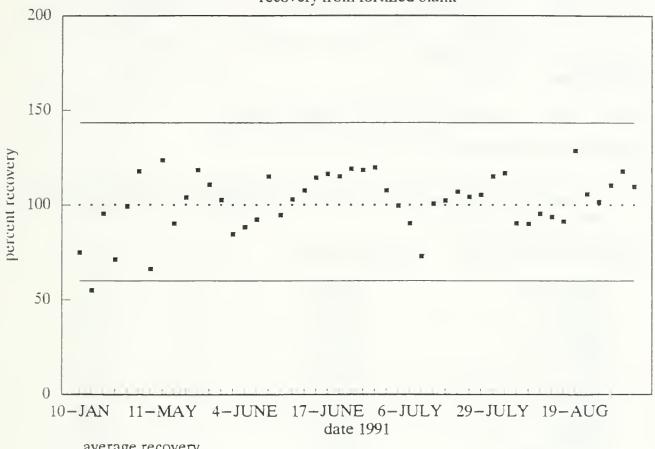




January - December 1991

Analyte	2,4-D (2,4-dichlorophenoxyacetic acid)
True Concentration	750 ng/L
Number of Observations	40
Within-run Standard Deviation	not available
Between-run Standard Deviation	20%
Accuracy (% of expected)	105%





January - December 1991

Analyte	Silvex
True Concentration	150 ng/L
Number of Observations	48
Within-run Standard Deviation	not available
Between-run Standard Deviation	16%
Accuracy (% of expected)	102%

METHOD CODE: PWAOP-E32224A.1

METHOD TITLE: The Determination of Organophosphorous Pesticides in Drinking Water by

GC-TSD

**LABORATORY:** Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

Samples are solvent-extracted; water is removed from extract and extract is evaporated to dryness. The reconstituted extract is examined by dual capillary gas chromatography with a thermionic specific detector.

PARAMETERS MEASURED:	LIS TEST CODE:	W ( ng/L )	T ( ng/L )
mathril trithian	DANGED I	200	2 000
methyl trithion	P4MTRI	200	2 000
dichlorvos	P4DICH	20	200
mevinphos	P4MEVI	20	200
phorate (thimet)	P4PHOR	20	200
diazinon	P4DIAZ	20	200
ronnel	P4RONN	20	200
chlorpyriphos (dursban)	P4DURS	20	200
reldan	P4RELD	20	200
malathion	P4MALA	20	200
parathion	P4PARA	20	200
methyl parathion	P4MPAR	20	200
ethion	P4ETHI	20	200

#### **REPORTING FORMAT:**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS:** During the period starting January 1991 and ending December 1991, a total of 24 method blanks was prepared and tested by the method. For these 24 analyses, no observable responses of any of the target analytes were encountered.

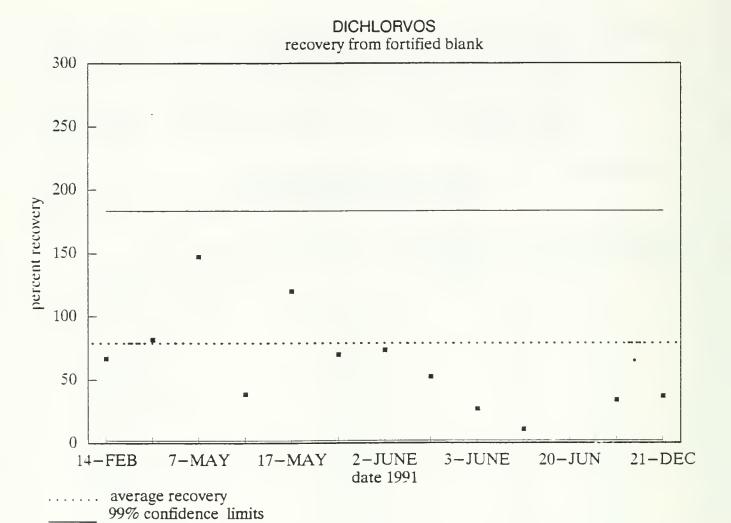
In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts: Dichlorvos (recovery from fortified blank)

Diazinon ( recovery from fortified blank ) Ethion ( recovery from fortified blank )

List of Performance Tables: Dichlorvos

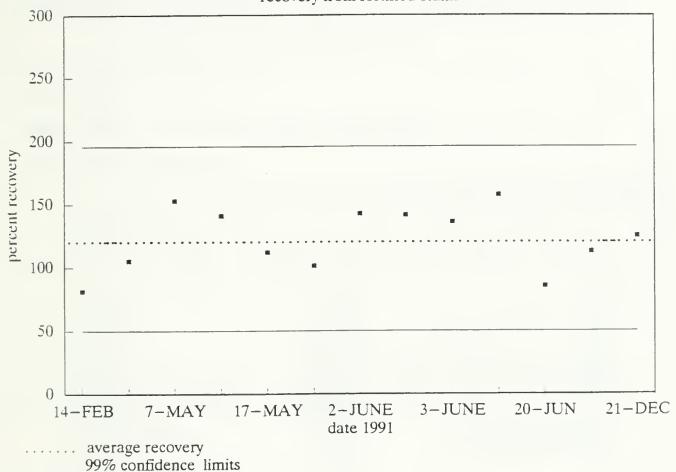
Diazinon Ethion



January - December 1991

Analyte	Dichlorvos
True Concentration	100 ng/L
Number of Observations	12
Within-run Standard Deviation	not available
Between-run Standard Deviation	33%
Accuracy (% of expected)	81%

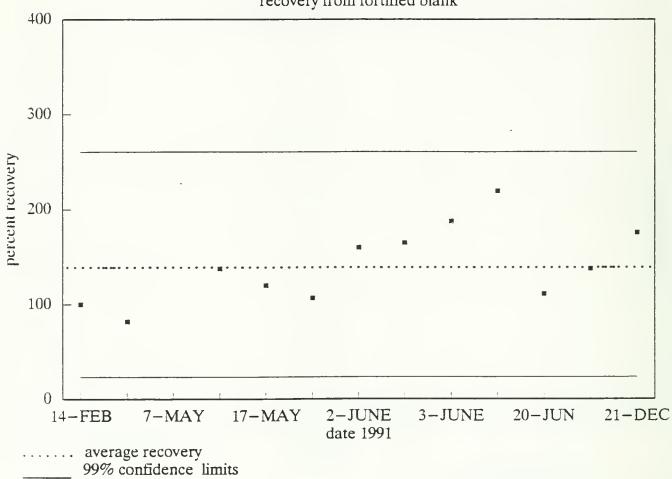




January - December 1991

Analyte	Diazinon
True Concentration	100 ng/L
Number of Observations	13
Within-run Standard Deviation	not available
Between-run Standard Deviation	23%
Accuracy (% of expected)	122%





January - December 1991

Analyte	Ethion
True Concentration	200 ng/L
Number of Observations	13
Within-run Standard Deviation	not available
Between-run Standard Deviation	39%
Accuracy (% of expected)	141%

METHOD CODE: HPLC/L-E3086A.1

**METHOD TITLE:** The Determination of Polynuclear Aromatic Hydrocarbons in Surface Water,

Drinking Water and Groundwater by HPLC

LABORATORY: Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, drinking water

#### PRINCIPLE OF THE METHOD:

Sample is solvent-extracted; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography equipped with fluorescence detector.

LIS TEST CODE:	W ( ng/L )	T ( ng/L )
B3001X	10	100
B3002X	1	10
B3003X	20	200
B3004X	20	200
B3005X	20	200
B3006X	50	500
B3007X	5	50
B3008X	50	500
B3010X	10	100
B3011X	10	100
B3012X	1	10
B3013X	5	50
B3014X	20	200
B3015X	10	100
B3016X	20	200
B3017X	2	20
B3019X	10	100
	B3001X B3002X B3003X B3004X B3005X B3006X B3007X B3008X B3010X B3011X B3012X B3012X B3013X B3014X B3015X B3016X B3017X	B3001X 10 B3002X 1 B3003X 20 B3004X 20 B3005X 20 B3006X 50 B3007X 5 B3008X 50 B3010X 10 B3011X 10 B3011X 10 B3011X 10 B3012X 1 B3013X 5 B3014X 20 B3015X 10 B3015X 10 B3017X 2

#### REPORTING FORMAT:

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS:** During the period starting January 1991 and ending December 1991, a total of 91 method blanks was prepared and tested by the method. For these 91 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts: Phenanthrene (recovery from fortified blank)

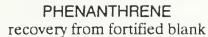
Benzo(b)fluoranthene / Perylene ( recovery from fortified blank )

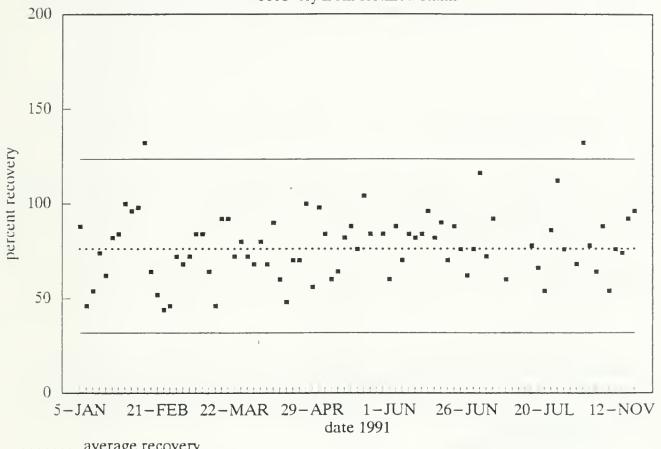
Benzo(a)pyrene (recovery from fortified blank)

List of Performance Tables: Phenanthrene

Benzo(b)fluoranthene / Perylene

Benzo(a)pyrene



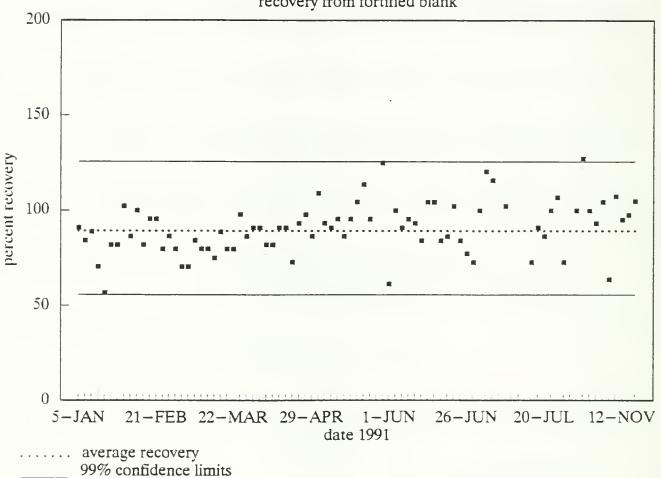


Performance Summary Table

January - December 1991

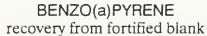
Analyte	phenanthrene
True Concentration	50 ng/L
Number of Observations	81
Within-run Standard Deviation	not available
Between-run Standard Deviation	18%
Accuracy (% of expected)	78%

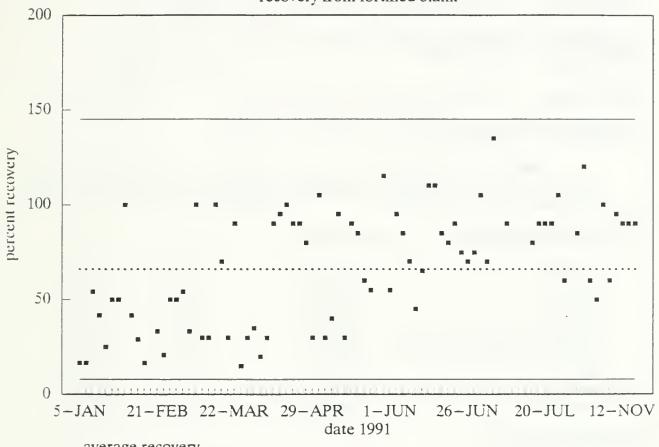




January - December 1991

Analyte	benzo(b)fluoranthene/perylene	
True Concentration	44 ng/L; 40 ng/L	
Number of Observations	81	
Within-run Standard Deviation	not available	
Between-run Standard Deviation	13%	
Accuracy (% of expected)	90%	





Performance Summary Table

January - December 1991

Analyte	benzo(a)pyrene
True Concentration	24 ng/L; 20ng/L
Number of Observations	80
Within-run Standard Deviation	not available
Between-run Standard Deviation	30%
Accuracy (% of expected)	68%

**METHOD CODE:** PWACAR-E3158A.1

**METHOD TITLE:** The Determination of Carbamates in Water by HPLC

LABORATORY: Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, drinking water

#### PRINCIPLE OF THE METHOD:

Sample is solvent-extracted; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography, using a variable wavelength ultraviolet detector.

PARAMETERS MEASURED: LIS	TEST CODE :	W ( ng/L )	T ( ng/L )
carbofuran	P6CARB	2 000	20 000
carbaryl	P6SEVN	200	2 000
butylate	P6SUTN	2.000	20 000
propoxur	P6PROP	2 000	20 000
isopropyl-3-chlorophenyl carbamate	P6CIPC	2 000	20 000
isopropyl phenyl carbamate	P6IPC	2 000	20 000
bux	P6BUX	200	2 000
diallate	P6DIAL	2 000	20 000
eptam	P6EPTM	2 000	20 000

#### **REPORTING FORMAT:**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

**REMARKS:** During the period starting January 1991 and ending December 1991, a total of 16 method blanks was prepared and tested by the method. For these 16 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts: Carbaryl (recovery from fortified blank)

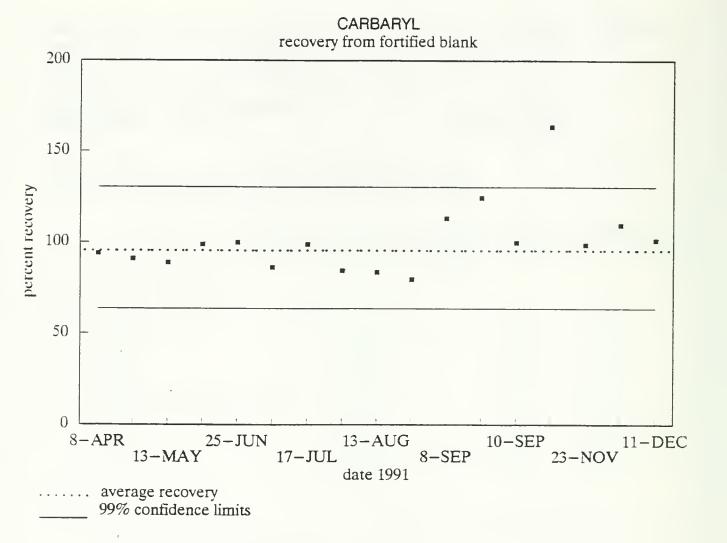
Isopropyl-3-chlorophenyl Carbamate (recovery from fortified blank)

Butylate (recovery from fortified blank)

List of Performance Tables: Carbaryl

Isopropyl-3-chlorophenyl Carbamate

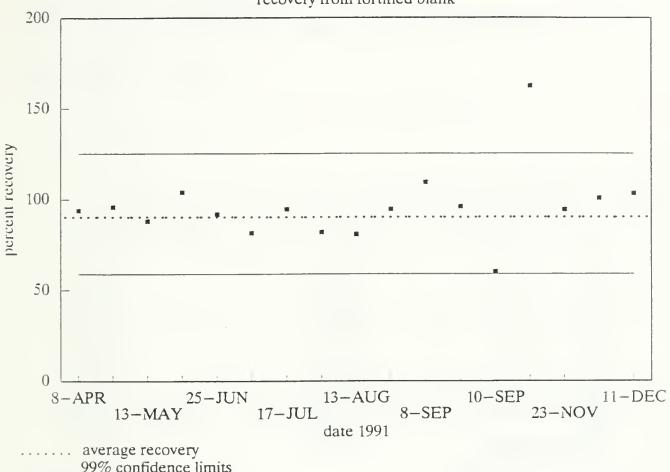
Butylate



January - December 1991

Analyte	carbaryl	
True Concentration	10 000 ng/L	
Number of Observations	15	
Within-run Standard Deviation	not available	
Between-run Standard Deviation	11%	
Accuracy (% of expected)	97%	

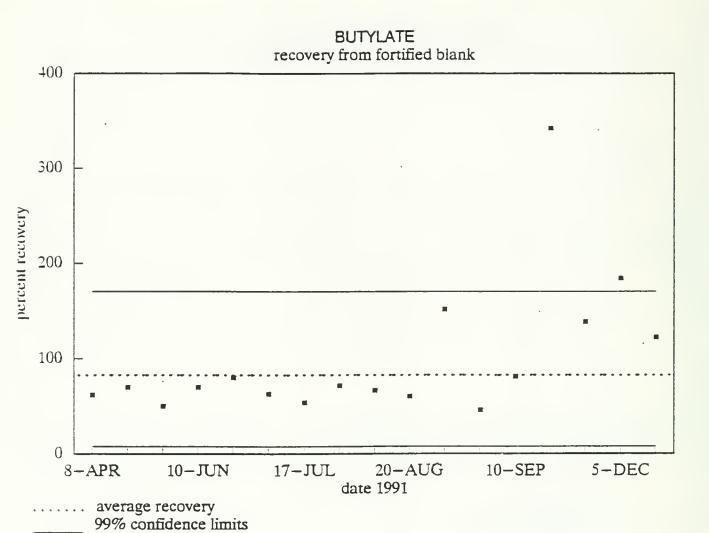
# ISOPROPYL-3-CHLOROPHENYL CARBAMATE recovery from fortified blank



99% confidence limits

January - December 1991

Analyte	CIPC (isopropyl-3-chlorophenyl carbamate)
True Concentration	5 000 ng/L
Number of Observations	15
Within-run Standard Deviation	not available
Between-run Standard Deviation	11%
Accuracy (% of expected)	92%



Performance Summary Table

January - December 1991

Analyte	butylate (Sutan)
True Concentration	5 000 ng/L
Number of Observations	15
Within-run Standard Deviation	not available
Between-run Standard Deviation	31%
Accuracy (% of expected)	79%

)

METHOD CODE: OWTRI-E3121A.1

**METHOD TITLE:** The Determination of Triazine Herbicides in Water by GC-TSD

LABORATORY: Organic Water SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

#### PRINCIPLE OF THE METHOD:

Samples are extracted with solvent, the extract is dried and then evaporated to dryness. The reconstituted extract is examined by gas chromatography using a thermionic specific detector.

PARAMETERS MEASURED:	LIS TEST CODE :	<b>W</b> ( ng/L )	T ( ng/L )
prometone	P2PROM	50	500
atraton	P2ATRO	50	500
propazine	P2PROP	50	500
atrazine	P2ATRA	50	500
prometryne	P2PROY	50	500
simazine	P2SIM	50	500
ametryne	P2AMET	50	500
sencor	P2SENC	100	1 000
bladex	P2BLAD	100	1 000
metolachlor	P0MET	500	5 000
alachlor	POLASS	500	5 000

#### REPORTING FORMAT:

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

**REMARKS:** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts: Atrazine (recovery from fortified blank)

Bladex (recovery from fortified blank)

Metolachlor (recovery from fortified blank)

List of Performance Tables: Method Blanks Summary

Atrazine Bladex Metolachlor

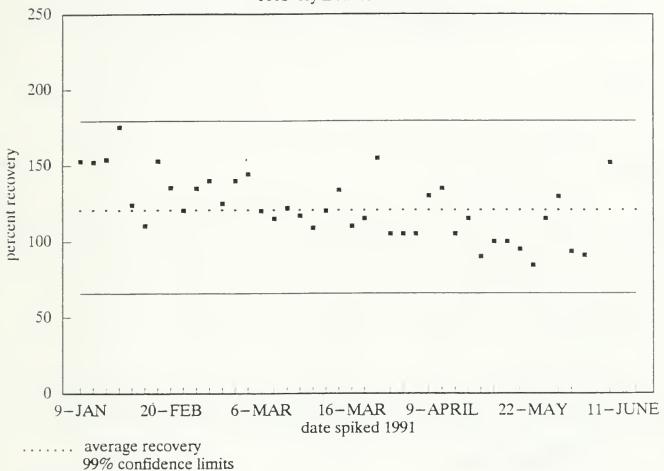
Method Blanks Summary

January 1991 - December 1991

1170diod Diminio			
Analyte	Number of Observations	Average Concentration ( ng/L )	Standard Deviation ( ng/L )
prometone	84	0	0
atraton	84	0	0
propazine	84	0	0
atrazine	84	25 *	51
prometryne	84	0	0
simazine	84	0	0
ametryne	84	0	0
sencor	84	0	0
bladex	84	0	0
metolachlor	84	0	0
alachlor	84	0	0

The trace levels of atrazine were found in method blanks prepared from tap water. In June 1991, Nanopure<sup>TM</sup> water started to be used for method blanks. No observable concentrations of atrazine were found in any of the total of 38 Nanopure<sup>TM</sup> water method blanks which were prepared and analyzed in the period starting June 1991 and ending December 1991.

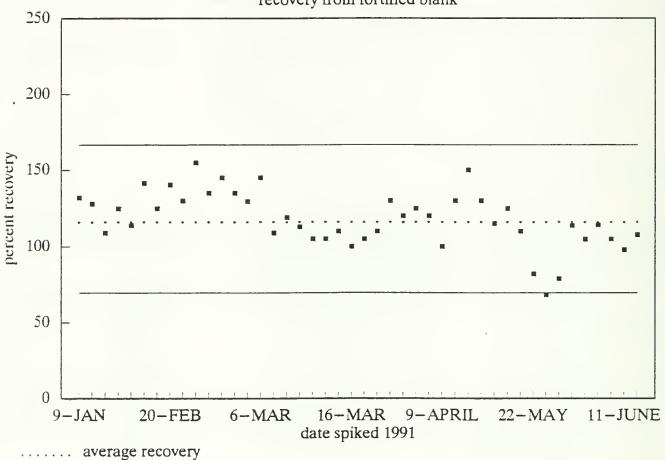
ATRAZINE recovery from fortified blank



January - December 1991

Analyte	atrazine
True Concentration	200 ng/L
Number of Observations	41
Within-run Standard Deviation	not available
Between-run Standard Deviation	21%
Accuracy (% of expected)	122%

BLADEX recovery from fortified blank

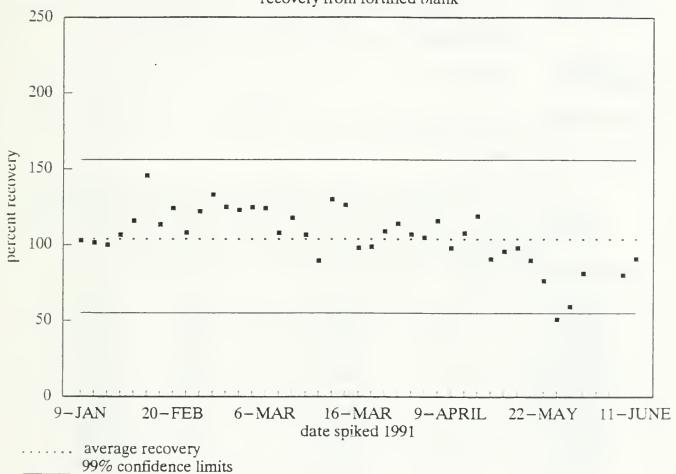


...... average recovery
99% confidence limits

January - December 1991

Analyte	bladex
True Concentration	200 ng/L
Number of Observations	44
Within-run Standard Deviation	not available
Between-run Standard Deviation	18%
Accuracy (% of expected)	118%

# METOLACHLOR recovery from fortified blank



\_\_\_\_\_ *JJ70* confidence mints

Performance Summary Table

Analyte	metolachlor
True Concentration	1 000 ng/L
Number of Observations	41
Within-run Standard Deviation	not available
Between-run Standard Deviation	19%
Accuracy (% of expected)	106%

METHOD CODE: PWAUH-E3230A.1

METHOD TITLE: The Determination of Phenyl Ureas in Water by High Performance Liquid

Chromatography

LABORATORY: Organic Water Unit

SUPERVISOR: C.D. Hall

**SAMPLE TYPE:** surface water, groundwater, finished drinking water

# PRINCIPLE OF THE METHOD:

Samples are extracted with an organic solvent; the extract is filtered through granular anhydrous sodium sulphate to remove water and evaporated to dryness by rotary evaporator. The reconstitued extract is examined by high performance liquid chromatography using a variable wavelength ultraviolet detector.

PARAMETERS MEASURED:	LIS TEST CODE :	<b>W</b> ( ng/L )	T ( ng/L )
linuron	P5LINU	2 000	20 000
monuron	P5MONU	2 000	20 000
diuron	P5DIUR	2 000	20 000
chlortoluron	P5CTOL	2 000	20 000
fluometuron	P5FMET	2 000	20 000
monolinuron	P5MLIN	2 000	20 000
chlorbromuron	P5CBRO	2 000	20 000
metoxuron	P5METX	2 000	20 000
siduron	P5SID	2 000	20 000
difenoxuron	P5DIF	2 000	20 000
neburon	P5NEB	2 000	20 000
paratan	P5PATO	2 000	20 000

### **REPORTING FORMAT:**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of 100 ng/L and up to maximum of two significant figures.

# QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

**REMARKS:** During the period starting January 1991 and ending December 1991, a total of 3 method blanks was prepared and tested by the method. For these 3 analyses, no observable responses of any of the target analytes were encountered.

Since this method is not used on regular basis (less than 50 samples are analyzed per year), no control charts are maintained.

List of Performance Charts: not applicable

List of Performance Tables: Recoveries of Target Analytes from Fortified Method Blanks

# Performance Summary Table

# Recoveries of PWAUH Target Analytes from Fortified Method Blanks

Analyte	concentration ( ng/L )	number of obs.	accuracy ( % of expected )	standard deviation (%)
metoxuron	5 000	3	89%	7
monuron	5 000	3	91%	2
chlortoluron	5 000	3	102%	10
fluometuron	5 000	3	105%	12
diuron	5 000	3	106%	10
monolinuron	5 000	3	101%	10
difenoxuron	5 000	3	101%	12
metobromuron	5 000	3	104%	7
siduron	5 000	3	96%	12
linuron	5 000	3	104%	10
chlorbromuron	5 000	3	105%	10
neburon	5 000	3	100%	11

METHOD CODE: PAAFD-E3123A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and

Polychlorinated Dibenzofurans (PCDF) in Ambient Air

LABORATORY: Dioxin Unit SUPERVISOR: E. Reiner

**SAMPLE TYPE:** ambient air

## PRINCIPLE OF THE METHOD:

Samples are collected using a MOE-modified high-volume air sampler with a polyurethane foam (PUF) plug and Teflon-coated glass fiber filter paper. PCDDs and PCDFs are extracted from the PUF and filter paper using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove the bulk of the sample matrix and potential chemical interferences. After cleanup, the extract is evaporated to dryness.

The reconstituted extract is examined by gas chromatography - triple quadrupole tandem mass spectrometry (GC-MS-MS) or gas chromatography - high resolution mass spectrometry (GC-HRMS).

#### **PARAMETERS MEASURED:**

total tetrachlorinated dibenzo-p-dioxins ( TCDD )

total pentachlorinated dibenzo-p-dioxins (PCDD)

total hexachlorinated dibenzo-p-dioxins (HxCDD) total heptachlorinated dibenzo-p-dioxins (HpCDD)

total octachlorinated dibenzo-p-dioxins ( PCDD )

total tetrachlorinated dibenzofurans ( TCDF )

total pentachlorinated dibenzofurans ( PCDF )

total hexachlorinated dibenzofurans (HxCDF)

total heptachlorinated dibenzofurans ( HpCDF )

total octachlorinated dibenzofurans (OCDF)

2,3,7,8-tetrachlorodibenzo-p-dioxin

1,2,3,7,8-pentachlorodibenzo-p-dioxin

three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins

1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin

2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-pentachlorodibenzofuran

1,2,3,7,8-pentachlorodibenzofuran

four 2,3,7,8-substituted hexachlorodibenzofurans

( parameters measured continued )

1,2,3,4,6,7,8-heptachlorodibenzofuran

1.2.3.4.7.8.9-heptachlorodibenzofuran

1.2.3.4.6.7.8.9-octachlorodibenzofuran

# REPORTING FORMAT:

Results are reported as pg/m³ rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 0.001 pg/m³ to 0.01 pg/m³.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes ( at least one per each congener group ) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, results are reported uncorrected for internal standard recovery.

**REMARKS:** The performance of the method was examined through CCME Interlaboratory Study PCDD/PCDF in Ambient Air.

Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts: <sup>13</sup>C<sub>12</sub>-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)

<sup>13</sup>C<sub>12</sub>-Pentachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Hexachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Heptachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin (recovery of internal standard)

List of Performance Tables: Method Blanks Summary

<sup>13</sup>C<sub>12</sub>-2,3,7,8-Tetrachlorodibenzo-p-dioxin
 <sup>13</sup>C<sub>12</sub>-1,2,3,7,8-Pentachlorodibenzo-p-dioxin
 <sup>13</sup>C<sub>12</sub>-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
 <sup>13</sup>C<sub>12</sub>-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin

<sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin

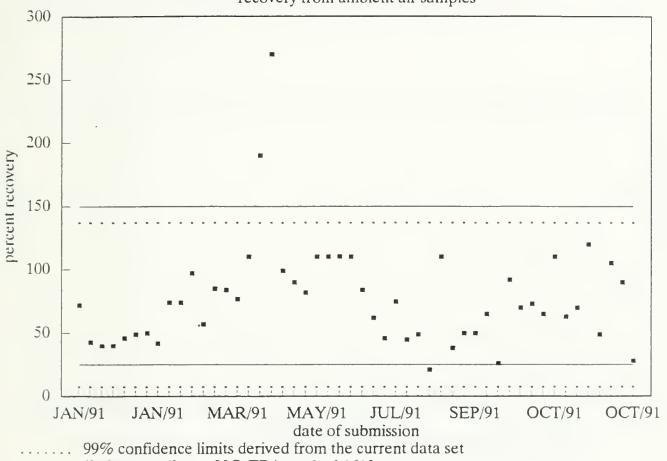
The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

Analyte	Number of Observations	Average Concentration (x10 <sup>-3</sup> pg/m <sup>3</sup> )	Standard Deviation (x10 <sup>-3</sup> pg/m <sup>3</sup> )
total TCDD total PCDD total HxCDD total HpCDD total OCDD	9 9 9 9	0 0 0.9 0 7.4	0 0 2.5 0 8.8
total TCDF total PCDF total HxCDF total HpCDF total OCDF	9 9 9 9	0 0 0 0	0 0 0 0





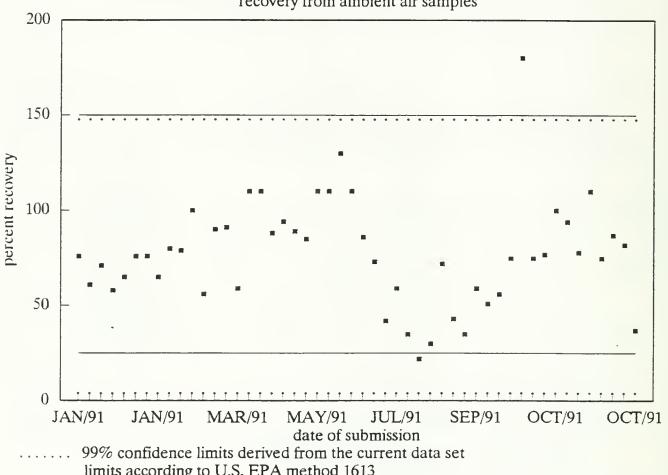
limits according to U.S. EPA method 1613

Performance Summary Table

Analyte ( Internal Standard )	<sup>13</sup> C <sub>12</sub> -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	1 pg/m³ °
Number of Observations	50
Within-run Rel. Standard Deviation	9% ( n=7 )
Between-run Standard Deviation	26%
Accuracy (% of expected)	71%

true concentration relates to the original sample volume of 3 000 m3; see official text of the method for the details on spiking procedure





limits according to U.S. EPA method 1613

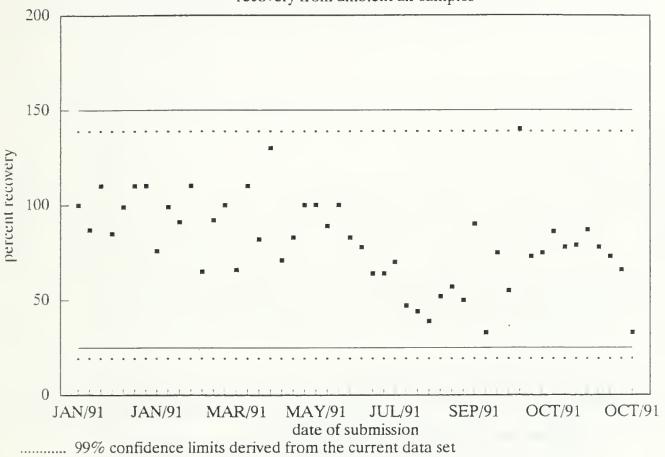
Performance Summary Table

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	1 pg/m³ *
Number of Observations	50
Within-run Rel. Standard Deviation	19% ( n=7 )
Between-run Standard Deviation	28%
Accuracy (% of expected)	77%

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

# 13-C-12-HEXACHLORODIBENZO-P-DIOXIN recovery from ambient air samples



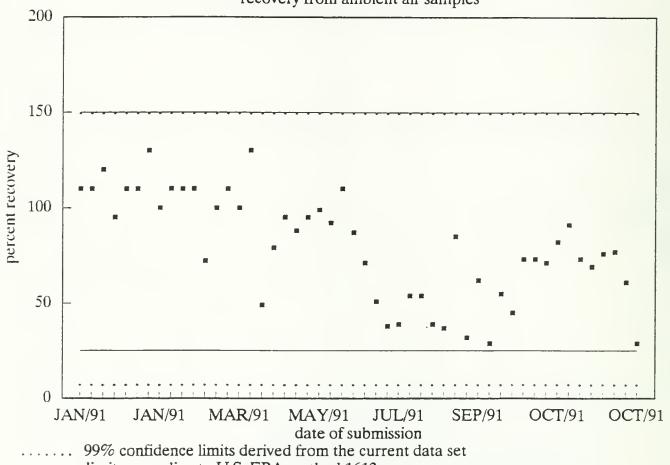
limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	
True Concentration	1 pg/m³ °	
Number of Observations	50	
Within-run Rel. Standard Deviation	9% ( n=7 )	
Between-run Standard Deviation	23%	
Accuracy (% of expected)	81%	

true concentration relates to the original sample volume of 3 000 m3; see official text of the method for the details on spiking procedure





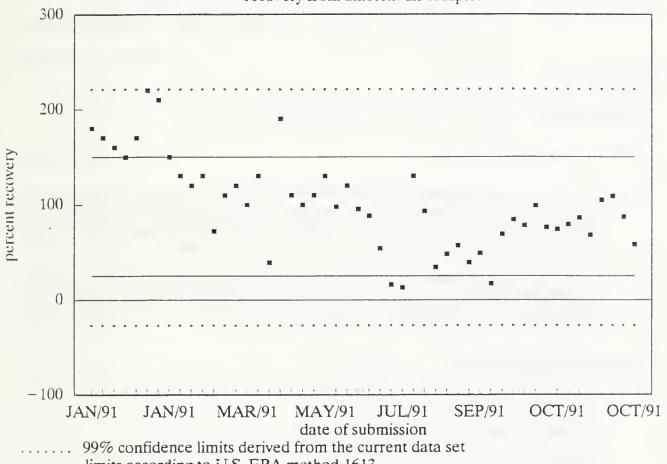
limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	
True Concentration	4 pg/m <sup>3</sup> *	
Number of Observations	50	
Within-run Rel. Standard Deviation	10% ( n=7 )	
Between-run Standard Deviation	28%	
Accuracy (% of expected)	80%	

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

# 13-C-12-OCTACHLORODIBENZO-P-DIOXIN recovery from ambient air samples



limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -octachlorodibenzo-p-dioxin	
True Concentration	3 pg/m³ °	
Number of Observations	50	
Within-run Rel. Standard Deviation	18% ( n=7 )	
Between-run Standard Deviation	48%	
Accuracy (% of expected)	100%	

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

**METHOD CODE:** PFAFD-E3135A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated

Dibenzofurans in Fish Tissue

LABORATORY: Dioxin Unit SUPERVISOR: E. Reiner

**SAMPLE TYPE:** fish tissue and other biological tissue (clams, shrimps)

#### PRINCIPLE OF THE METHOD:

Sample is homogenized by mechanical grinding. A homogeneous portion to be analyzed is fortified with isotopically labelled internal standard and is digested overnight with concentrated hydrochloric acid. The resulting solution is extracted with hexane and the extract is passed through a column containing anhydrous sodium sulphate and sulphuric acid-silica packing.

The extract is concentrated and subsequently fractionated using high performance liquid chromatography (HPLC). The reconstituted final extract is analyzed by gas chromatography - mass spectrometry or gas chromatography - triple quadrupole tandem mass spectrometry or gas chromatography - high resolution mass spectrometry.

#### **PARAMETERS MEASURED:**

total tetrachlorinated dibenzo-p-dioxins ( TCDD ) total pentachlorinated dibenzo-p-dioxins ( PCDD ) total hexachlorinated dibenzo-p-dioxins ( HxCDD ) total heptachlorinated dibenzo-p-dioxins ( HpCDD ) total octachlorinated dibenzo-p-dioxins ( OCDD ) total tetrachlorinated dibenzo-furans ( TCDF ) total pentachlorinated dibenzo-furans ( PCDF ) total hexachlorinated dibenzo-furans ( HxCDF ) total heptachlorinated dibenzo-furans ( HpCDF )

total octachlorinated dibenzofurans ( OCDF )

2,3,7,8-tetrachlorodibenzo-p-dioxin

1,2,3,7,8-pentachlorodibenzo-p-dioxin

three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins

1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin

2.3.7.8-tetrachlorodibenzofuran

2,3,4,7,8-pentachlorodibenzofuran

1,2,3,7,8-pentachlorodibenzofuran

four 2.3.7.8-substituted hexachlorodibenzofurans

1,2,3,4,6,7,8-heptachlorodibenzofuran

( parameters measured continued )

1,2,3,4,7,8,9-heptachlorodibenzofuran 1,2,3,4,6,7,8,9-octachlorodibenzofuran

#### REPORTING FORMAT:

Results are reported as ppt (picograms of CDD/CDF per gram of wet fish tissue) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 1 pg/g to 10 pg/g.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported uncorrected for internal standard recovery.

**REMARKS:** Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts: <sup>13</sup>C<sub>12</sub>-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)

<sup>13</sup>C<sub>12</sub>-Pentachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Hexachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Heptachlorodibenzo-p-dioxin (recovery of internal standard)
 <sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin (recovery of internal standard)

List of Performance Tables: Method Blanks Summary

 $^{13}$ C<sub>12</sub>-2,3,7,8-Tetrachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,7,8-Pentachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin

<sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin

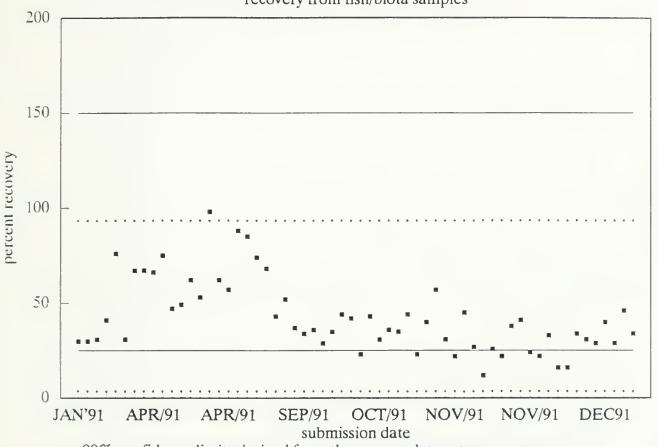
The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

Analyte	Number of Observations	Average Concentration ( pg/g )	Standard Deviation ( pg/g )
total TCDD total PCDD total HxCDD total HpCDD total OCDD	6 6 6 6	0.33 0.17 0 0.17 0.33	0.47 0.37 0 0.37 0.47
total TCDF total PCDF total HxCDF total HpCDF total OCDF	6 6 6 6	0.17 0.17 0 0 0	0.37 0.37 0 0 0

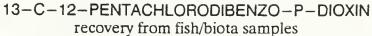
# 13-C-12-TETRACHLORODIBENZO-P-DIOXIN recovery from fish/biota samples

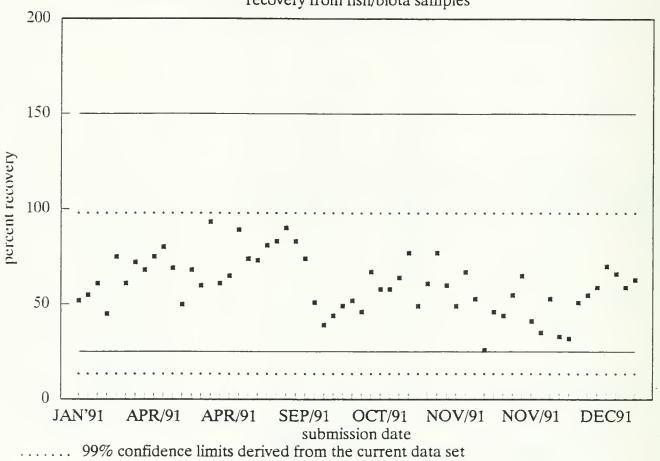


..... 99% confidence limits derived from the current data set limits according to U.S.EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -2,3,7,8-tetrachlorodibenzo-p-dioxin	
True Concentration	200 pg/g	
Number of Observations	60	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	19%	
Accuracy (% of expected)	43%	



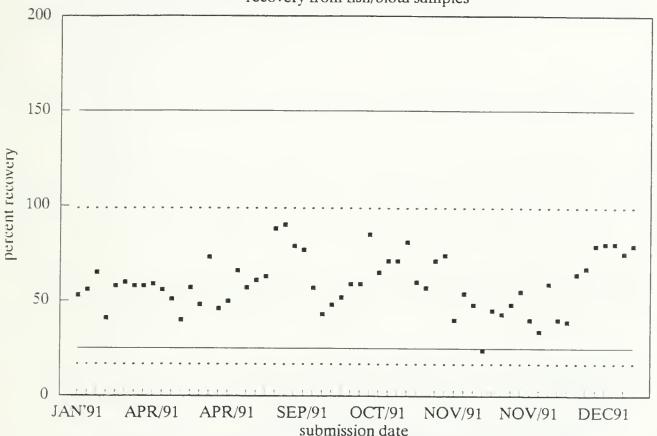


limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-pentachlorodibenzo-p-dioxin	
True Concentration	200 pg/g	
Number of Observations	60	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	15%	
Accuracy (% of expected)	61%	

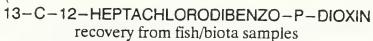


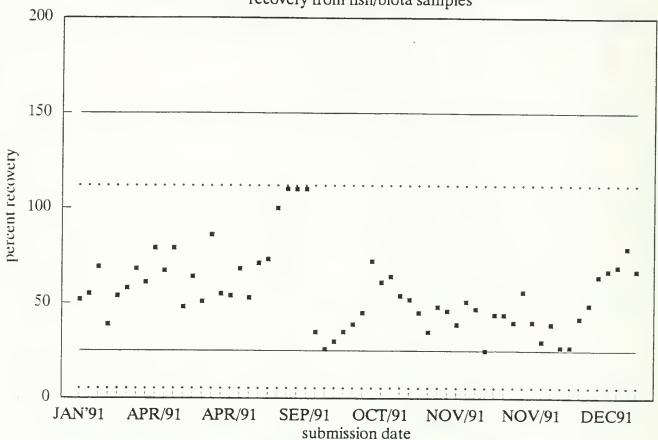


99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	
True Concentration	170 pg/g	
Number of Observations	60	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	20%	
Accuracy (% of expected)	59%	



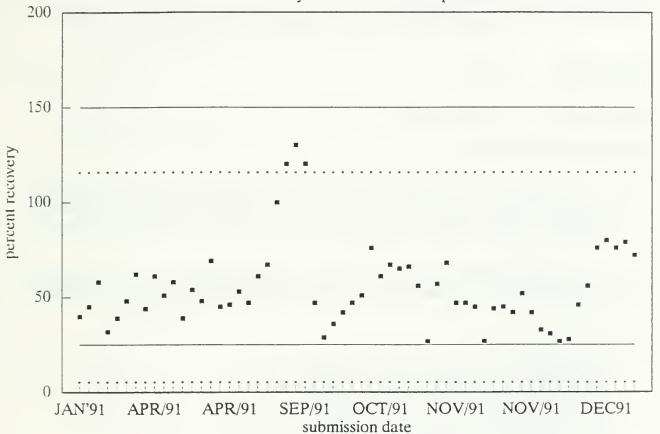


99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	
True Concentration	600 pg/g	
Number of Observations	60	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	14%	
Accuracy (% of expected)	56%	





..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -octachlorodibenzo-p-dioxin	
True Concentration	400 pg/g	
Number of Observations	60	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	22%	
Accuracy (% of expected)	56%	

METHOD CODE: PSAFD-E3152A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and

Polychlorinated Dibenzofurans (PCDF) in Soil and Sediment

LABORATORY: Dioxin Unit SUPERVISOR: E. Reiner

**SAMPLE TYPE:** soil and sediment

# PRINCIPLE OF THE METHOD:

Samples are dried, ground and homogenized. PCDD and PCDF are extracted from soil/sediment using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove bulk of the sample matrix and potential chemical interferences.

The reconstituted final extract is analyzed by gas chromatography - mass spectrometry or gas chromatography - triple quadrupole tandem mass spectrometry or gas chromatography - high resolution mass spectrometry.

### **PARAMETERS MEASURED:**

total tetrachlorinated dibenzo-p-dioxins (TCDD)

total pentachlorinated dibenzo-p-dioxins ( PCDD )

total hexachlorinated dibenzo-p-dioxins (HxCDD)

total heptachlorinated dibenzo-p-dioxins (HpCDD)

total octachlorinated dibenzo-p-dioxins (OCDD)

total tetrachlorinated dibenzofurans (TCDF)

total pentachlorinated dibenzofurans ( PCDF )

total hexachlorinated dibenzofurans (HxCDF)

total heptachlorinated dibenzofurans ( HpCDF )

total octachlorinated dibenzofurans (OCDF)

2,3,7,8-tetrachlorodibenzo-p-dioxin

1,2,3,7,8-pentachlorodibenzo-p-dioxin

three 2.3.7.8-substituted hexachlorodibenzo-p-dioxins

1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin

2.3.7.8-tetrachlorodibenzofuran

2.3.4.7.8-pentachlorodibenzofuran

1,2,3,7,8-pentachlorodibenzofuran

four 2,3,7,8-substituted hexachlorodibenzofurans

1,2,3,4,6,7,8-heptachlorodibenzofuran

1,2,3,4,7,8,9-heptachlorodibenzofuran

1,2,3,4,6,7,8,9-octachlorodibenzofuran

#### REPORTING FORMAT:

Results are reported as ppt (picograms of CDD/CDF per gram of soil) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 1 pg/g to 10 pg/g.

# QUALITY CONTROL:

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported uncorrected for internal standard recovery.

**REMARKS:** Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts: <sup>13</sup>C<sub>12</sub>-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)

 $^{13}\mathrm{C}_{12}\text{-Pentachlorodibenzo-p-dioxin}$  ( recovery of internal standard )  $^{13}\mathrm{C}_{12}\text{-Hexachlorodibenzo-p-dioxin}$  ( recovery of internal standard )  $^{13}\mathrm{C}_{12}\text{-Heptachlorodibenzo-p-dioxin}$  ( recovery of internal standard )  $^{13}\mathrm{C}_{12}\text{-Octachlorodibenzo-p-dioxin}$  ( recovery of internal standard )

List of Performance Tables: Method Blanks Summary

 $^{13}$ C<sub>12</sub>-2,3,7,8-Tetrachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,7,8-Pentachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin

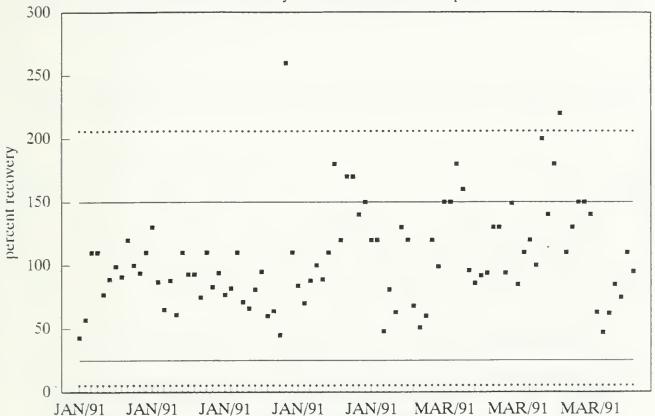
<sup>13</sup>C<sub>12</sub>-Octaehlorodibenzo-p-dioxin

The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary January 1991 - December 1991

Analyte	Number of Observations	Average Concentration ( pg/g )	Standard Deviation ( pg/g )
total TCDD	22	0	0
total PCDD	22	0.27	0.69
total HxCDD	22	0.27	1.3
total HpCDD	22	0.09	0.42
total OCDD	22	0.36	0.48
total TCDF	22	0.05	0.21
total PCDF	22	0.05	0.21
total HxCDF	22	0.14	0.34
total HpCDF	22	0.27	0.54
total OCDF	22	0.18	0.39





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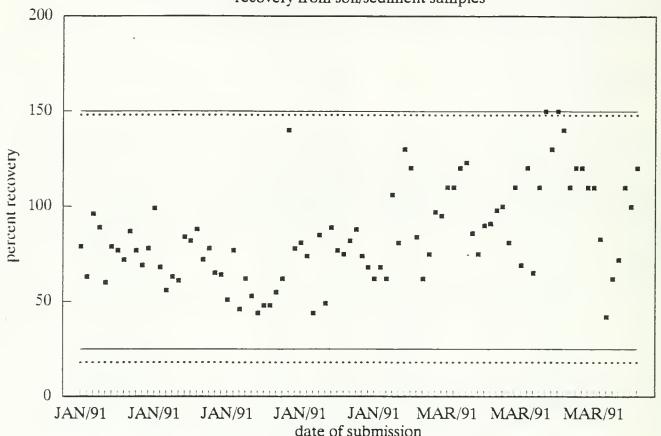
date of submission

..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -2,3,7,8-tetrachlorodibenzo-p-dioxin	
True Concentration	400 pg/g	
Number of Observations	92	
Within-run Rel. Standard Deviation	17% ( n=7 )	
Between-run Standard Deviation	39%	
Accuracy (% of expected)	106%	



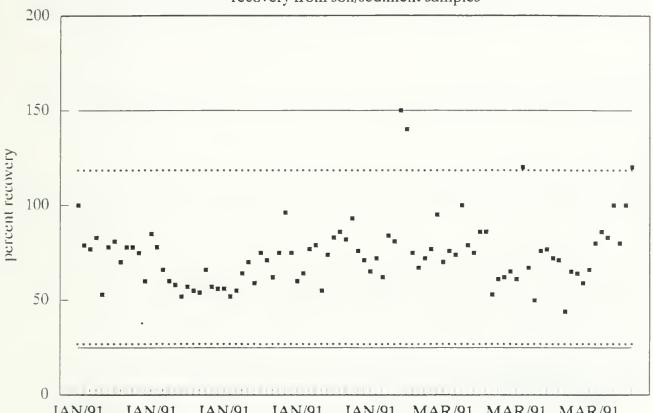


..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-pentachlorodibenzo-p-dioxin	
True Concentration	400 pg/g	
Number of Observations	92	
Within-run Rel. Standard Deviation	11% ( n=7 )	
Between-run Standard Deviation	25%	
Accuracy (% of expected)	85%	

# 13-C-12-HEXACHLORODIBENZO-P-DIOXIN recovery from soil/sediment samples



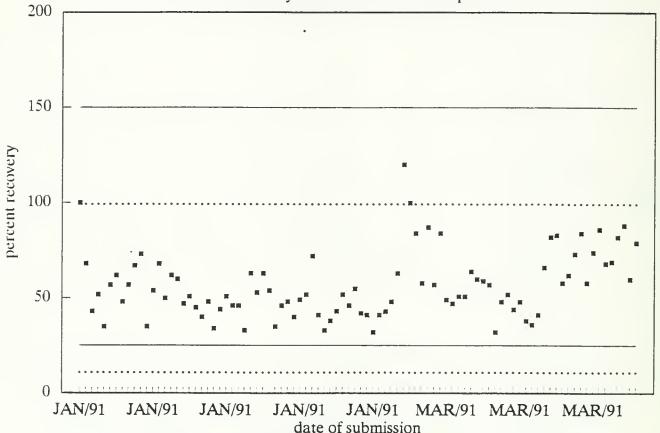
JAN/91 JAN/91 JAN/91 JAN/91 MAR/91 MAR/91 MAR/91 date of submission

..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	350 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	3% ( n=7 )
Between-run Standard Deviation	18%
Accuracy (% of expected)	72%



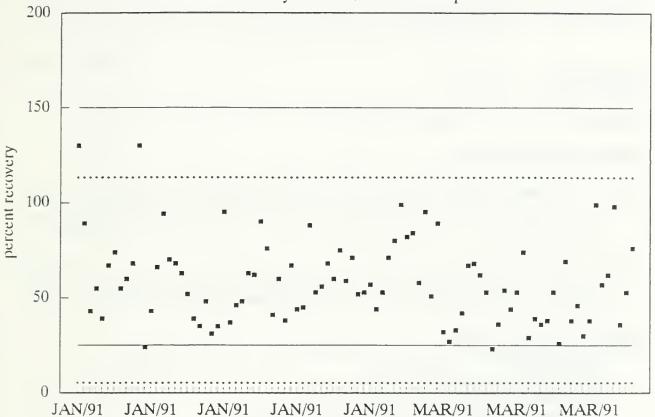


..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	1 200 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	17% ( n=7 )
Between-run Standard Deviation	17%
Accuracy (% of expected)	57%





MAR/91 MAR/91 MAR/91 date of submission

99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	$^{13}C_{12}$ -octachlorodibenzo-p-dioxin
True Concentration	800 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	6% ( n=7 )
Between-run Standard Deviation	22%
Accuracy (% of expected)	59%

METHOD CODE:

PWAFD-E3163A.1

METHOD TITLE:

The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated

Dibenzofurans in Drinking Water by GC-MS

LABORATORY: SUPERVISOR:

Dioxin Unit E. Reiner

SAMPLE TYPE :

raw or finished drinking water

## PRINCIPLE OF THE METHOD:

Target analyte(s) are extracted from samples with an organic solvent. The extract is then cleaned of potential chemical interferences by two-stage column chromatography. Cleaned extract is evaporated to dryness. After reconstitution, the extract is examined by gas chromatography mass spectrometry.

If the extract contains chemical interferences that prevent the quantification of target analytes, it is further fractionated using high performance liquid chromatography and then re-analyzed by GC-MS.

#### **PARAMETERS MEASURED:**

total tetrachlorinated dibenzo-p-dioxins total pentachlorinated dibenzo-p-dioxins total hexachlorinated dibenzo-p-dioxins total heptachlorinated dibenzo-p-dioxins total octachlorinated dibenzo-p-dioxins total tetrachlorinated dibenzo-p-dioxins total pentachlorinated dibenzo-furans total pentachlorinated dibenzo-furans total hexachlorinated dibenzo-furans total heptachlorinated dibenzo-furans

2,3,7,8-tetrachlorodibenzo-p-dioxin

total octachlorinated dibenzofurans

1,2,3,7,8-pentachlorodibenzo-p-dioxin

three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins

1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin

2.3.7.8-tetrachlorodibenzofuran

2,3,4,7,8-pentachlorodibenzofuran

1,2,3,7,8-pentachlorodibenzofuran

four 2,3,7,8-substituted hexachlorodibenzofurans

1,2,3,4,6,7,8-heptachlorodibenzofuran

1,2,3,4,7,8,9-heptachlorodibenzofuran

1.2.3.4.6.7.8.9-octachlorodibenzofuran

#### REPORTING FORMAT:

Results are reported in parts per quadrillion (pg/L) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 1 ppq to 5 ppq.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the relevant results are reported uncorrected for internal standard recovery.

**REMARKS:** During the period starting January 1991 and ending December 1991, a total of ten method blanks was prepared and tested by the method. For these 10 analyses, no observable responses of any of the target analytes were encountered.

Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts: <sup>13</sup>C<sub>12</sub>-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)

 $^{13}$ C<sub>12</sub>-Pentachlorodibenzo-p-dioxin (recovery of internal standard)  $^{13}$ C<sub>12</sub>-Hexachlorodibenzo-p-dioxin (recovery of internal standard)  $^{13}$ C<sub>12</sub>-Heptachlorodibenzo-p-dioxin (recovery of internal standard)  $^{13}$ C<sub>12</sub>-Octachlorodibenzo-p-dioxin (recovery of internal standard)

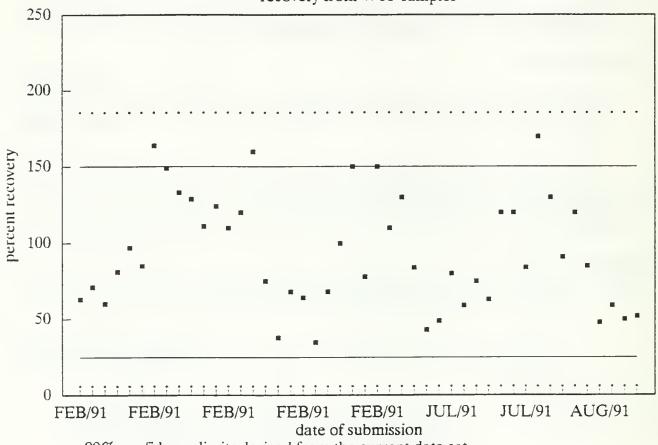
List of Performance Tables: <sup>13</sup>C<sub>12</sub>-2,3,7,8-Tetrachlorodibenzo-p-dioxin

<sup>13</sup>C<sub>12</sub>-1,2,3,7,8-Pentachlorodibenzo-p-dioxin
 <sup>13</sup>C<sub>12</sub>-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
 <sup>13</sup>C<sub>12</sub>-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin

<sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin

The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.



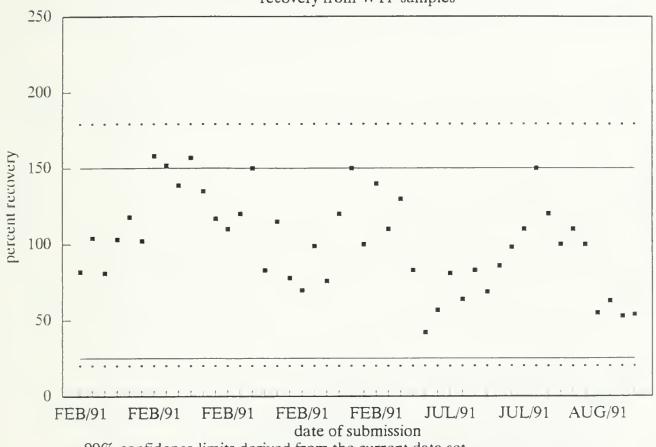


...... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	1 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	16%
Accuracy (% of expected)	57%



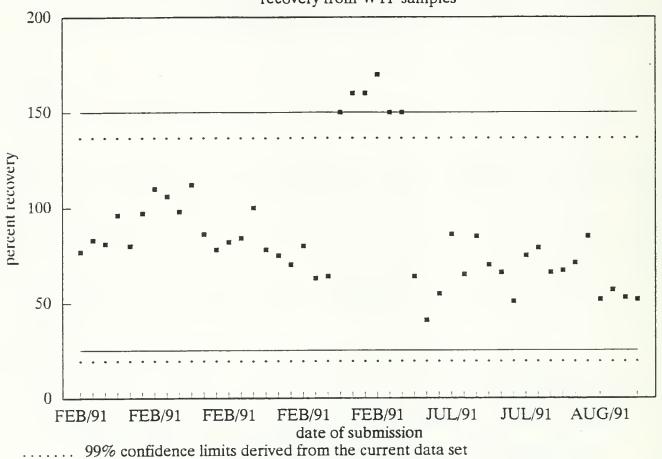


... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-pentachlorodibenzo-p-dioxin	
True Concentration	1 ng/L	
Number of Observations	117	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	17%	
Accuracy (% of expected)	72%	



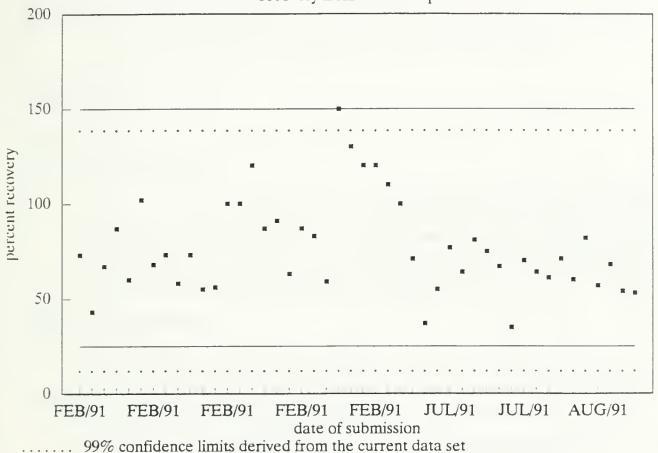


...... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	
True Concentration	0.8 ng/L	
Number of Observations	117	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	21%	
Accuracy (% of expected)	76%	

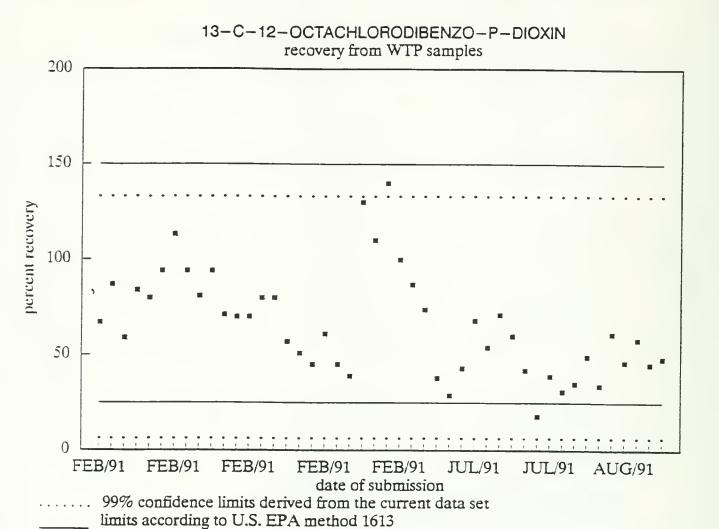




..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	
True Concentration	3 ng/L	
Number of Observations	117	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	22%	
Accuracy (% of expected)	73%	



## Performance Summary Table

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -octachlorodibenzo-p-dioxin	
True Concentration	2 ng/L	
Number of Observations	117	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	21%	
Accuracy (% of expected)	69%	

METHOD CODE: PWAFD-E3164A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated

Dibenzofurans in Groundwater and Aqueous Effluent by GC-MS

LABORATORY: Dioxin Unit SUPERVISOR: E. Reiner

**SAMPLE TYPE:** groundwater, aqueous industrial or municipal effluent

#### PRINCIPLE OF THE METHOD:

Sample is filtered to remove visible particulates; the aqueous and particulate portions are processed separately. Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo-furans are extracted from each portion; Soxhlet apparatus is used for filtered particulate portion extraction. Both extracts are dried, concentrated and cleaned up using two-stage chromatographic columns. After clean-up, extracts are evaporated to dryness. The reconstituted extracts are examined by gas chromatography - mass spectrometry.

#### PARAMETERS MEASURED:

total tetrachlorinated dibenzo-p-dioxins (TCDD) total pentachlorinated dibenzo-p-dioxins (PCDD)

total hexachlorinated dibenzo-p-dioxins (HxCDD)

total heptachlorinated dibenzo-p-dioxins (HpCDD)

total octachlorinated dibenzo-p-dioxins (OCDD)

total tetrachlorinated dibenzofurans ( TCDF )

total pentachlorinated dibenzofurans ( PCDF )

total hexachlorinated dibenzofurans ( HxCDF )

total heptachlorinated dibenzofurans ( HpCDF )

total octachlorinated dibenzofurans ( OCDF )

2.3.7.8-tetrachlorodibenzo-p-dioxin

1,2,3,7,8-pentachlorodibenzo-p-dioxin

three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins

1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin

2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-pentachlorodibenzofuran

1.2.3.7.8-pentachlorodibenzofuran

four 2,3,7,8-substituted hexachlorodibenzofurans

1,2,3,4,6,7,8-heptachlorodibenzofuran

1,2,3,4,7,8,9-heptachlorodibenzofuran

1.2.3.4.6.7.8.9-octachlorodibenzofuran

### **REPORTING FORMAT:**

Results are reported in parts per quadrillion (pg/L) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 5 ppq to 10 ppq.

#### QUALITY CONTROL:

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes ( at least one per each congener group ) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported not corrected for recovery of internal standard.

**REMARKS:** Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts: <sup>13</sup>C<sub>12</sub>-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)

 $^{13}C_{12}^{-1}$ -Pentachlorodibenzo-p-dioxin ( recovery of internal standard )  $^{13}C_{12}$ -Hexachlorodibenzo-p-dioxin ( recovery of internal standard )  $^{13}C_{12}$ -Heptachlorodibenzo-p-dioxin ( recovery of internal standard )

<sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin (recovery of internal standard)

List of Performance Tables: Method Blanks Summary

 $^{13}$ C<sub>12</sub>-2,3,7,8-Tetrachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,7,8-Pentachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin  $^{13}$ C<sub>12</sub>-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin

<sup>13</sup>C<sub>12</sub>-Octachlorodibenzo-p-dioxin

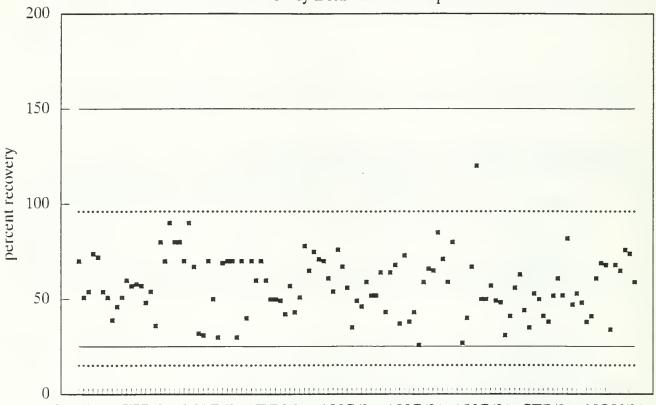
The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

Analyte	Number of Observations	Average Concentration ( pg/L )	Standard Deviation ( pg/L )
total TCDD	23	0	0
total PCDD	23	0.05	0.21
total HxCDD	23	0.18	0.65
total HpCDD	23	0	0
total OCDD	23	0.18	0.83
total TCDF	23	0	0
total PCDF	23	0	0
total HxCDF	23	0.05	0.21
total HpCDF	23	0.05	0.21
total OCDF	23	0	0

## 13-C-12-TETRACHLORODIBENZO-P-DIOXIN recovery from effluent samples

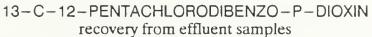


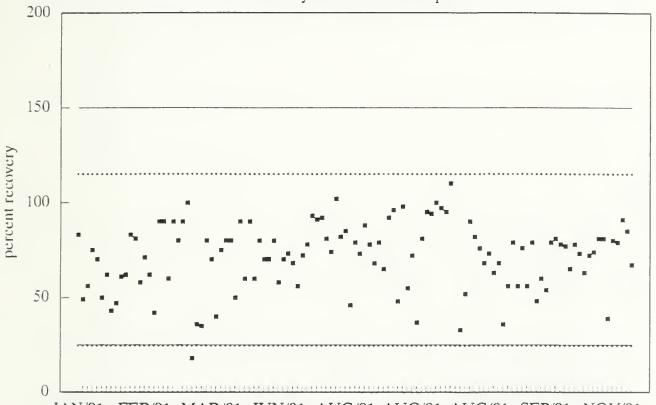
JAN/91 FEB/91 MAR/91 JUN/91 AUG/91 AUG/91 AUG/91 SEP/91 NOV/91 date of submission

..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -2,3,7,8-tetrachlorodibenzo-p-dioxin	
True Concentration	350 pg/L	
Number of Observations	46	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	36%	
Accuracy (% of expected)	94%	





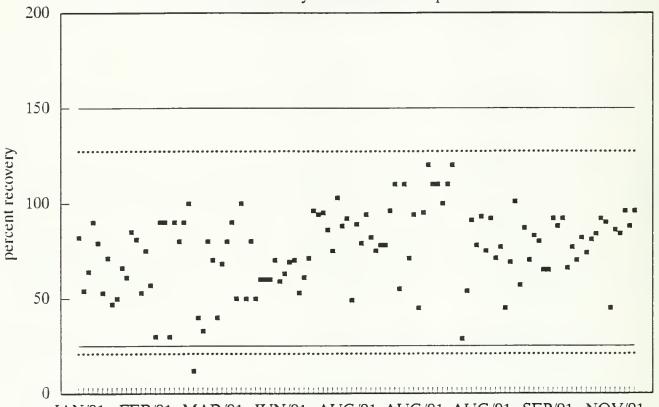
JAN/91 FEB/91 MAR/91 JUN/91 AUG/91 AUG/91 AUG/91 SEP/91 NOV/91 date of submission

99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-pentachlorodibenzo-p-dioxin	
True Concentration	350 pg/L	
Number of Observations	46	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	31%	
Accuracy (% of expected)	102%	

## 13-C-12-HEXACHLORODIBENZO-P-DIOXIN recovery from effluent samples



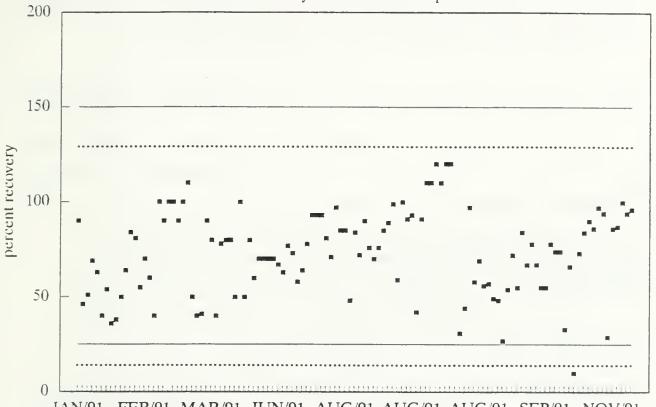
JAN/91 FEB/91 MAR/91 JUN/91 AUG/91 AUG/91 AUG/91 SEP/91 NOV/91 date of submission

...... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	
True Concentration	300 pg/L	
Number of Observations	46	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	23%	
Accuracy (% of expected)	80%	

# 13-C-12-HEPTACHLORODIBENZO-P-DIOXIN recovery from effluent samples



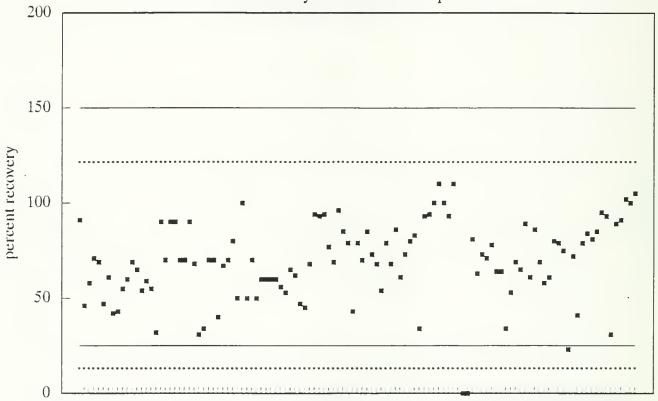
JAN/91 FEB/91 MAR/91 JUN/91 AUG/91 AUG/91 AUG/91 SEP/91 NOV/91 date of submission

..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

Analyte (Internal Standard)	1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	
True Concentration	1 000 pg/L	
Number of Observations	46	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	25%	
Accuracy (% of expected)	77%	

# 13-C-12-OCTACHLORODIBENZO-P-DIOXIN recovery from effluent samples



JAN/91 FEB/91 MAR/91 JUN/91 AUG/91 AUG/91 AUG/91 SEP/91 NOV/91 date of submission

..... 99% confidence limits derived from the current data set limits according to U.S. EPA method 1613

Performance Summary Table

January - December 1991

Analyte (Internal Standard)	<sup>13</sup> C <sub>12</sub> -octachlorodibenzo-p-dioxin	
True Concentration	600 pg/L	
Number of Observations	46	
Within-run Rel. Standard Deviation	not available	
Between-run Standard Deviation	26%	
Accuracy (% of expected)	66%	

METHOD CODE :

NDMA-E3291A.1

METHOD TITLE:

The Determination of N-Nitrosodimethylamine (NDMA) in Drinking Water and in Aqueous Samples by Gas Chromatography / High Resolution Mass

Spectrometry (GC/HRMS)

LABORATORY:

Mass Spectrometry Unit

SUPERVISOR:

V. Taguchi

SAMPLE TYPE:

drinking water, aqueous samples

### PRINCIPLE OF THE METHOD:

Samples which contain particulate matter are filtered prior to analytical processing.

Sample pH is adjusted to 12 to keep the acidic components in the aqueous phase and the resulting solution is serially extracted with dichloromethane. The dichloromethane extract is washed with a sulphuric acid solution to remove basic components from the organic phase. After being filtered through granular anhydrous sodium sulphate to remove water the extract is concentrated by rotary evaporator and a nitrogen evaporating unit.

The final extract containing the remaining neutral components is analyzed by GC/HRMS. NDMA is quantified by an isotope dilution method.

PARAMETERS MEASURED:

LIS TEST CODE:

MDL (µg/L)

N-Nitrosodimethylamine

MSOBNO (NDMA)

0.005

#### REPORTING FORMAT:

Results are reported in µg/L rounded off to two significant figures. The lowest reported value is  $5x10^3\mu g/L$ .

## QUALITY CONTROL:

The routine quality control operations monitor validity of calibration (repetitive analysis of calibration standard), size of potential positive bias (method blanks) and maintenance of required instrument sensitivity.

**REMARKS**: The performance of the method was examined through performance audit samples program and and MOE Interlaboratory Study # 91-2. Both programs were organized by LSB Quality Management Office.

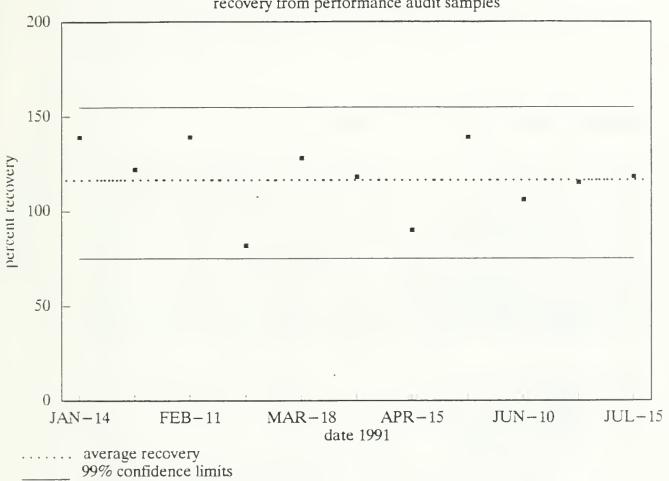
List of Performance Charts: N-Nitrosodimethylamine (recovery from performance audit samples)

N-Nitrosodimethylamine ( results for method blanks )

List of Performance Tables: N-Nitrosodimethylamine (intralaboratory and interlaboratory performance

summary )

## N-NITROSODIMETHYLAMINE recovery from performance audit samples



Performance Summary Table

January - December 1991

performance audit samples MSU reproducibility study

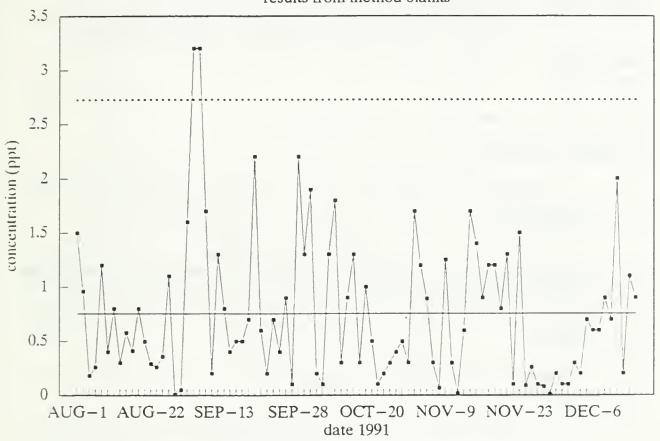
Analyte	N-nitrosodimethylamine	
True Concentration	(0.075 - 0.100) μg/L	69 μg/L
Number of Observations	12	10
Within-run Rel. Standard Deviation	not available	7.5%
Between-run Standard Deviation	18%	19%
Accuracy (% of expected)	118%	156%

## Inter-laboratory Comparisons

## MOE Inter-laboratory study # 91-2

Number of Participants	13	13
Number of Samples	2	2
Design Value	0.302 μg/L	0.588 μg/L
Inter-laboratory Mean	0.423 μg/L	0.682 μg/L
Inter-laboratory R.S.D.	20%	22%
Intra-laboratory Mean	0.300 μg/L	0.640 μg/L
Intra-laboratory R.S.D.	0%	8%

## N-NITROSODIMETHYLAMINE results from method blanks



average concentration found (ppt)
...... upper 95%—confidence limit derived from the current data set

Method Blanks Summary

August - December 1991

Analyte	N-nitrosodimethylamine		
Number of Observations	92		
Mean Concentration	0.76x10 <sup>-3</sup> μg/L		
Standard Deviation	0.67x10 <sup>-3</sup> μg/L		

METHOD CODE:

SMY-E3186A.1

METHOD TITLE:

The Determination of Extractable Organics in Drinking Water, Aqueous Samples,

Soil and Sediment by Gas Chromatography / Mass Spectrometry (GC/MS)

LABORATORY:

Mass Spectrometry Unit

SUPERVISOR:

V. Taguchi

**SAMPLE TYPE:** 

drinking water, aqueous samples, soil, sediment

## PRINCIPLE OF THE METHOD:

The method involves extraction of the organic components from their respective matrices with an organic solvent and analysis of the concentrated extract by gas chromatography / full scan mass spectrometry.

Drinking water and aqueous samples are initially adjusted to pH 12. The base/neutral components are serially extracted with dichloromethane. The remaining aqueous fraction is then adjusted to pH 2 and the acidic components are serially extracted with dichloromethane. The extracts are filtered through anhydrous sodium sulphate to remove water and then concentrated by rotary evaporator and a nitrogen evaporating unit. The final extracts are analyzed by GC/FS-MS.

Soil and sediment samples are extracted with toluene in a Soxhlet/Dean-Stark apparatus. The concentrated extracts are analyzed by GC/FS-MS.

The extractable organics are reported according to their compound names and compound classes. The concentrations of the components are approximate and are calculated relative to the internal standard.

## **PARAMETERS MEASURED:**

LIS TEST CODE:

extractable organics analyzable by GC/MS

**PBEXT** 

### REPORTING FORMAT:

The extractable organics are reported according to their compound names, CAS registry numbers and compound classes  $^{\circ}$ . The concentrations of all components are approximate and are reported in  $\mu g/L$  ( resp. ng/g for soil and sediment samples ) to one significant figure with the prefix "A-". The minimum reported concentrations (calculated relatively to the internal standard ) depend on sample matrix and range from  $0.1~\mu g/L$  to  $1~\mu g/L$ .

## QUALITY CONTROL:

The routine quality control operations monitor presence of potential interferences in the method blanks, relative extraction efficiencies ( surrogates ), instrumental performance ( MS tuning characteristics / autotune /, reference standard solution analysis ).

The ratio of  $d_6$ -NDMA to the internal standard  $d_{10}$ -phenanthrene is used to monitor extraction efficiencies (recoveries) of a polar compound relative to a non-polar compound and is also an indicator for chromatographic performance.

**REMARKS:** The performance of the method was periodically examined through performance audit samples program administered by LSB Quality Management Office.

List of Performance Tables: Performance Audit Samples Summary

Clement, R.E.; Taguchi, V.Y.; Techniques for the Gas Chromatography - Mass Spectrometry Identification of Organic Compounds in Effluents; Environment Ontario, July 1989

## Performance Audit Samples Summary, January - December 1991

analyte	number of tests	concentration range (µg/L)	analyte identified [percent of tests]	recovery within (10-200)% (% of identified)
bis(2-chloroethyl) ether	20	3.0 - 6.0	20 [100%]	100 %
1,3-dichlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
1,2-dichlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
N-nitroso-di-n-propylamine	20	3.0 - 6.0	19 [95%]	95 %
isophorone	20	3.0 - 6.0	16 [80%]	100 %
bis(2-chloroethoxy)methane	20	3.0 - 6.0	20 [100%]	100 %
1,2,4-trichlorobenzene	20	3.0 - 6.0	20 [100%]	95 %
hexachlorobutadiene	20	3.0 - 6.0	18 [90%]	100 %
2-chloronaphthalene	20	3.0 - 6.0	18 [90%]	100 %
2,6-dinitrotoluene	20	3.0 - 6.0	18 [90%]	100 %
2,4-dinitrotoluene	20	3.0 - 6.0	20 [100%]	100 %
diethyl phthalate	20	3.0 - 6.0	20 [100%]	100 %
hexachlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
phenanthrene	20	3.0 - 6.0	20 [100%]	100 %
dibutyl phthalate	20	3.0 - 6.0	17 [85%]	100 %
pyrene	20	3.0 - 6.0	19 [95%]	100 %
benzo(a)anthracene	18	3.0 - 6.0	15 [83%]	100 %
dioctyl phthalate	20	3.0 - 6.0	16 [80%]	89 %
benzo(k)fluoranthene	20	3.0 - 6.0	16 [80%]	89 %
base/neutral extractables total	378		352 [93%]	98 %
phenol	20	1.9 - 4.0	19 [95%]	90 %
2-chlorophenol	20	2.5 - 5.5	20 [100%]	90 %
2,4,6-trichlorophenol	20	1.5 - 3.2	20 [100%]	95 %
p-chloro-m-cresol	20	2.5 - 5.0	19 [95%]	95 %
2-nitrophenol	20	2.0 - 4.0	15 [75%]	100 %
2,4-dichlorophenol	20	2.3 - 4.7	19 [95%]	100 %
4-nitrophenol	20	0.5 - 1.0	4 [20%]	75 %
2,4-dimethylphenol	20	3.5 - 7.7	14 [70%]	93 %
pentachlorophenol	20	3.0 - 6.0	13 [65%]	92 %
acidic extractables total	180		143 [79%]	94 %

METHOD CODE: SMY-E3189A.1

METHOD TITLE:

The Determination of Volatile Organics in Drinking Water and Aqueous Samples by Purge-and-Trap Gas Chromatography / Mass Spectrometry (P&T/GC/MS)

Mass Spectrometry Unit

LABORATORY: SUPERVISOR:

V. Taguchi

SAMPLE TYPE:

drinking water, aqueous samples

#### PRINCIPLE OF THE METHOD:

The volatile organics are purged from the matrix with helium gas, isolated onto multi-layered trap(s) and then thermally desorbed and analyzed by gas chromatography / full scan mass spectrometry.

The volatile organics are reported according to their compound names and compound classes. The concentrations of the components are approximate and are calculated relative to the internal standard.

#### PARAMETERS MEASURED:

LIS TEST CODE:

volatile organics analyzable by P&T/GC/MS

**PBVOL** 

## REPORTING FORMAT:

The volatile organics are reported according to their compound names, CAS registry numbers and compound classes. The concentrations of all components are approximate and are reported to one significant figure with the prefix "A-". The minimum reported concentrations (calculated relatively to the internal standard) depend on sample matrix and range from 0.1 µg/L to 1 µg/L.

## QUALITY CONTROL:

The routine quality control operations monitor the presence of potential interferences (blanks), relative purging efficiencies (surrogates), instrumental performance (MS tuning characteristics / autotune /, reference standard solution analysis).

Clement, R.E.; Taguchi, V.Y.; Techniques for the Gas Chromatography - Mass Spectrometry Identification of Organic Compounds in Effluents; Environment Ontario, July 1989

The ratio of  $d_4$ -1,2-dichloroethane to the internal standard is used to monitor relative purging efficiencies (recoveries) and is also an indicator for the relative response factors of the components. For MISA samples, 1,3-dichlorobutane is used as an internal standard, for all other samples, the internal standard is  $d_{10}$ -ethylbenzene.

**REMARKS:** The performance of the method was periodically examined through performance audit samples program administered by LSB Quality Management Office.

List of Performance Tables: Performance Audit Samples Summary

## Performance Audit Samples Summary, January - December 1991

analyte	number of tests	concentration range (µg/L)	analyte identified [percent of tests]	recovery within (50-150)% (% of identified)
1,2-dichloroethane	9	0.9 - 2.0	9 [100%]	100 %
chlcroform	9	6.5 - 15.0	9 [100%]	89 %
1,1,1-trichloroethane	9	0.6 - 1.4	9 [100%]	89 %
trichloroethylene	9	0.4 - 1.0	9 [100%]	100 %
carbon tetrachloride	9	1.3 - 3.0	9 [100%]	100 %
tetrachloroethylene	9	0.8 - 1.7	9 [100%]	100 %
bromodichloromethane	9	0.4 - 1.0	9 [100%]	100 %
dibromochloromethane	9	1.0 - 2.4	9 [100%]	100 %
bromoform	9	1.5 - 3.3	9 [100%]	100 %
o-xylene	9	0.9 - 2.0	9 [100%]	100 %
m&p-xylene	9	1.7 - 3.7	9 [100%]	100 %
1,2-dichlorobenzene	9	0.9 - 2.1	9 [100%]	100 %
1,3-dichlorobenzene	9	0.9 - 2.0	9 [100%]	100 %
1,4-dichlorobenzene	9	0.9 - 2.0	9 [100%]	100 %
1,2-dibromoethane	9	0.9 - 2.0	9 [100%]	100 %
volatile organics total	135		135 [100%]	98.5 %



## **GLOSSARY OF TERMS**

accuracy proximity to the true value expressed as average percent recovery or

average percent of expected

average (mean) sum of the measurements divided by the number of measurements

between-run experiment samples are prepared by different technicians, and the instrumental

analyses take place under different calibrations of the analytical system

between-run r.s.d. measure of reproducibility of a method

calibration solution solution containing target analyte(s) for a particular method at

concentration(s) that will produce response(s) falling within the linear range of the instrument. This solution is used to calibrate the instrument

response with respect to the analyte concentration.

calibration check solution solution which has composition similar to the calibration solution and

which is prepared independently of calibration solution. It is used to check performance of the instrument, especially the validity of current

calibration.

fortified method blank synthetic sample prepared by adding known quantities of target analytes

of the method to the interference-free matrix

internal standard known amount of a compound, that is assumed to have identical chemical

and physical properties with the analyte(s) of interest, is added to the sample prior to sample processing. The recovery of this compound from

the sample is used for correction of the final results.

MDL method detection limit. MDL marks the concentration level above which

one can conclude that a measured result indicates the presence of analyte

in the sample with a specified confidence (99%).

method code Analytical Methods Catalogue Code used within Ontario Ministry of the

Environment

percent recovery ratio of the concentration obtained by the experiment to the theoretical

concentration, multiplied by one hundred

performance charts graphical presentation of the individual results of the analyses of fortified

method blanks or internal standards. The x-axis on the chart represents the date, the y-axis outlines percent recovery. The average and 99%

confidence limits are displayed as well.

relative standard deviation measure of spread of a population. The square root of the squared sum

of the measurements minus the sum of squared measurements, divided by

the number of measurements minus one.

T value level below which analytical results represent trace values; additional data

are needed for valid interpretation ( see Code of Practice for Environmental Laboratories, September 1989, Ontario Ministry of the

Environment)

upper and lower 99% confi-  $UL(LL) = X + (-) t \times s$ 

dence limit, UL (LL) X,s represent the average and the standard deviation of the replicate

measurement:

t<sub>(n-1,α=0.01)</sub> is the Student's t-value appropriate for a 99% confidence level

and the given number of degrees of freedom n

within-run experiment samples are prepared and analyzed by a single technician, and the

instrumental analyses take place within one calibration of the analytical

system

within-run r. s. d. measure of repeatability of a method

W value minimum reported level ( see Code of Practice for Environmental

Laboratories, September 1989, Ontario Ministry of the Environment)



